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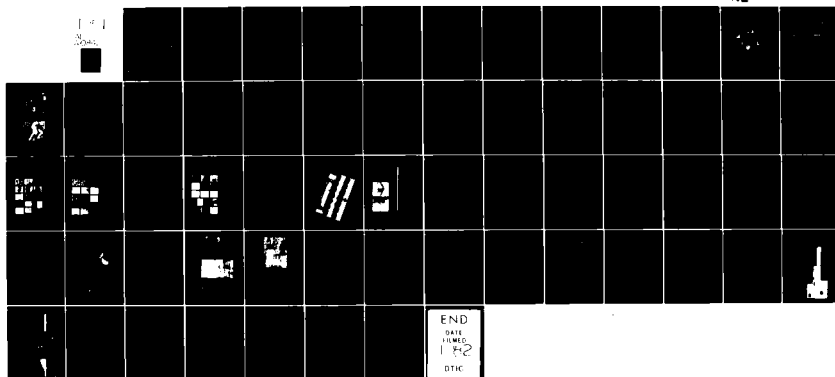   ELECTROTRANSPORT IN THIN FILMS.(U)
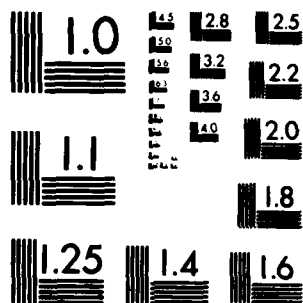   OCT 81 R E HUMMEL, R T DEHOFF   AFOSR-76-2963
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The objective of this research was to develop a scientific perspective that forms a foundation for understanding the processes that lead to the development of holes in thin film metal strips. This understanding allows the prediction and control of hole formation in thin film connectors that are an integral part of all microelectronic devices, thus extending their design lifetime, and improving their reliability. (over)

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This project focused upon the effects of dopants on the behavior of thin films subject to high current densities, and upon the relative contributions of the several mass transport processes that may contribute to changes in the structure of thin film strips. Specifically, in addition to electrotransport, which may involve both electron wind and electrolysis effects, grain boundary grooving, driven by surface tension forces, and thermotransport, driven by temperature gradients, was found to significantly alter the structure of the strip. Experimental strategies have been developed which emphasize each of these contributions, permitting assessment of their relative roles in the overall process.

Examination of the resulting samples with transmission optical, transmission electron and scanning electron microscopy revealed that 1) all three mechanisms may contribute significantly to hole formation in undoped films, 2) addition of indium stabilizes the as-deposited grain size, minimizes thermal grooving but promotes hole formation by the other two mechanisms, and 3) addition of sodium greatly enhances grain boundary grooving, promotes early failure in AC-stressed samples and results in hole formation at the anode under a DC.

The doping of gold films has been achieved by vapor deposition, by ion bombardment and by depositing the gold films on sodium lime silica glass, whereas the Na was picked up by the gold film during Joule heating. All three doping mechanisms lead to a reversal of the failure site in gold films during DC stressing. It was found that sodium constituents as low as 50 ppm still caused the failure site in gold to be on the anode side (reversal).

Research Objectives for the Contract Period

The objectives for this research have been expressed in the "Introduction" of our original proposal submitted to AFOSR in February, 1975. These lines are reproduced below:

Thin film technology is a classic case of a segment of industry in which the rapid development of the technology has outstripped the scientific understanding of the principles that govern the behavior of the product. For example, thin gold films are widely used as metallizations in microelectronic devices, and their use is increasing; yet, even the <u>direction</u> of electrotransport in gold films is a subject of intensive controversy. The electrotransport behavior of many metals potentially useful to the industry, such as silver, copper or gold, is largely un-investigated. The role of microstructure in electrotransport has been explored, but not established; it is not definitively known, for example, whether a coarse or fine grained film is desirable, or, indeed, whether it makes much difference at all.

Recent studies, in our laboratory and elsewhere, indicate that impurities may play an important role in electrotransport response. Such impurities may be inadvertently added, as in the case of a film deposited upon a soluble underlayer (gold on chromium, molybdenum, etc.), or the impurity may be purposely co-deposited in an attempt to improve resistance to electrotransport failure. Evidence for the role of impurities is rudimentary. No principles have been established for selecting candidate impurity additions, either on the basis of their electronic structure, or by analogy with their behavior in bulk systems.

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The primary reason for our lack of scientific knowledge in this field lies in the nature of industrial research, particularly in the last decade. The pressure to solve immediate problems, or to improve existing products, does not admit the systematic study of thin film behavior necessary to establish a scientific perspective. Such is the purpose of university research. Accordingly, this proposal lays out a research plan designed to clarify and resolve the controversies existing in the field, and to expand the scientific foundation in a systematic program.

Summary of Significant Accomplishments

The results of our research on "Electromigration in Thin Films"
in the contract period 1/1/76-8/31/81 have been published in three
scientific papers which appeared in refereed journals. These papers are
reproduced below. A preliminary draft of a fourth paper is also included
which summarizes our most recent research findings.

ELECTROMIGRATION IN THIN FILMS*

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Electrotransport in thin films can be observed at much lower temperatures than in bulk metals because of the good heat conductivity of the substrates on which the films are usually deposited, and because of the large surface to volume ratio. As a consequence, grain boundary or even surface transport takes place. This has been substantiated by activation energy measurements.

The influence of gradients in temperature, grain size, and structural parameters on the ion flux under the influence of a direct current of high density is discussed. These gradients lead to voids at one end and accumulation of material at the other end of the film. Impurities in the metal film cause a retardation of the ion flux, probably because of the segregation of these impurities on the grain boundaries. The influence of the substrate on electrotransport and the location of void and hillock formation is discussed.

Introduction

Electrotransport experiments in thin films can be conducted at substantially higher current densities than in bulk specimens. Current densities of the order of some 10^6 A/cm^2 can be easily achieved compared to 10^4 A/cm^2 in bulk materials. Nevertheless, because of the relatively good heat conductivities of the sub-strates on which these films are usually deposited, the temperature of thin films can be kept at low enough values to observe grain boundary (or even surface) transport. Thus, temperatures between 150 and 300°C are not uncommon in thin film electrotransport experiments.

A further peculiarity contributes to these relatively low temperatures: since the film thicknesses are typically a few 1000 Angstroms, whereas the stripes are still up to a few millimeters wide, the surface to volume ratio is large, which aids in the dissipation of heat, generated by the electric current in the film. Thin film electrotransport experiments are, therefore, ideally suited to complement bulk electrotransport studies by extending the useful temperature range to lower values. This shall be demonstrated in a few examples.

Grain Boundary and Surface Migration

The velocity of ions (v) which migrate under the influence of a direct current can be plotted in an Arrhenius-type diagram according to the equation

$$\ln \frac{v}{j} = \ln \frac{D_o \; eZ^* \; \rho}{kT} - \frac{Q}{k}\frac{1}{T}$$

(where j = current density, k = Boltzmann constant, T = temperature, D_o = pre-exponential diffusion constant, eZ* = effective charge and ρ = resistivity). From the slopes of these curves, the activation energies (Q) for the processes can be determined.

In Fig. 1, data for thin film electrotransport in aluminum, obtained between 220°C and 360°C are shown,[1] together with values for bulk electrotransport,[2] measured in the temperature range between 440°C and 590°C. It can be seen that the combined bulk and thin film electrotransport studies cover a wide range of temperatures, which one method alone would be able to achieve only with great difficulties.

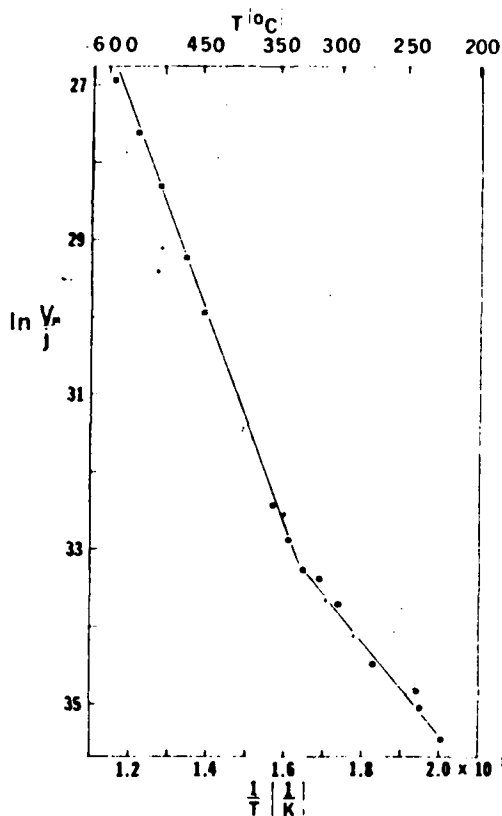From Fig. 1, an activation energy for electrotransport in aluminum of 1.16 ev[2,3] at temperatures

Fig. 1.   In (v_m/j) vs. $1/T$ (v_m = marker velocity) for
electrotransport in aluminum films ([●] un-
annealed films, [+] annealed films, [■] bulk
specimens). From Ref. 1 and 2.

above 330°C can be deduced which is approximately the
same value which was found in aluminum for lattice
self-diffusion (between 1.4 and 1.48 ev[4-6]). At lower
temperatures, an activation energy of 0.53 ev can be
obtained. This value is approximately one-half of the
above mentioned Q, found for bulk electromigration, a
result which is significant because it suggests that
below 330°C, grain boundary transport becomes dominant
in aluminum. This has been substantiated also by di-
rect observations, as we will see later.

A second example for how various transport mech-
anisms dominate in different temperatures ranges, elec-
trotransport in silver films will now be discussed. As
above, two distinct temperatures ranges are observed

in an appropriate Arrhenius plot (Fig. 2) displaying
markedly different activation energies: at temperatures
above 220°C, the activation energy (0.95 ev[7]) is ap-
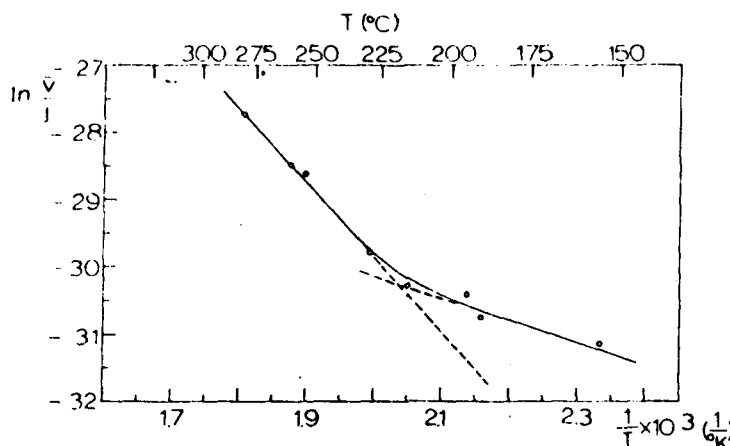proximately that which was found for grain boundary

Fig. 2. ln(v/j) vs. 1/T for electrotransport in silver
films (annealed). From Ref. 7.

self-diffusion (0.82 ev - 0.93 ev[8-12]). Below 220°C, a
smaller activation energy (0.3 ev) can be observed for
electrotransport in silver films.[7] This value is simi-
lar to the one measured for surface self-diffusion
(0.35 ev - 0.43 ev[13,14]).

These measurements clearly demonstrate that thin
film electrotransport is characterized as a rule by ion
migration in grain boundaries or, at lower tempera-
tures, even by migration on the surface of the metal
films.

Divergencies in Ion Flux Due to Temperature Gradients

Electrotransport in thin films attracted substan-
tial interest about ten years ago when it became evi-
dent that certain failures in metallizations of micro-
minaturized electrical circuits were due to ion migra-
tion under the influence of high current densities. In
order to obtain these failures, i.e., the divergencies

ih ion flux, it is a prerequisite that a gradient in
temperature, grain size, cross section or another para-
meter along the thin film stripe be present. Those
gradients are virtually always observed in practical
applications. This is particularly true for gross tem-
perature gradients which are caused by Joule heating of
the thin film stripes combined with cooling of the
thicker ends. As a consequence, higher temperatures at
the center of thin film conductors are obtained, as
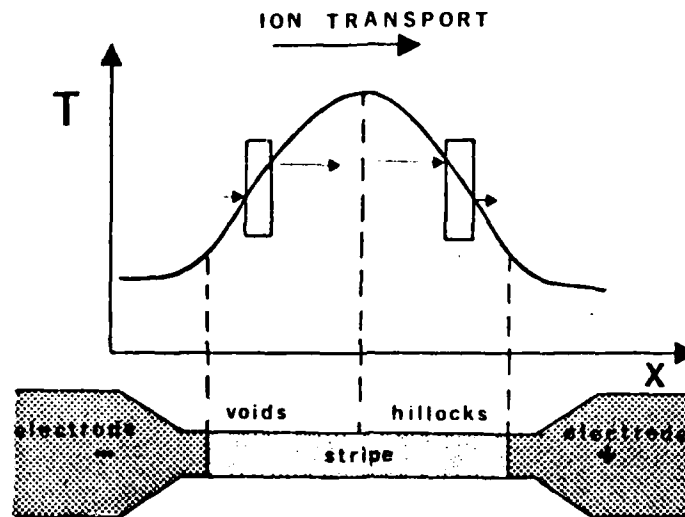schematically shown in Fig. 3. Because of such a tem-

Fig. 3. Schematic representation of a temperature
gradient (caused by Joule heating) along a
thin film stripe. The direction of ion trans-
port due to the "electron wind effect" is in-
dicated.

perature profile, more ions are leaving a given unit
volume at the cathode end than are entering it, which
results in a depletion of material and consequently
void formation. Similarly, more ions are entering a
unit volume close to the anode than leaving it, causing
accumulation of material. This observation is repre-
sented by the following equation which shows that the
largest divergence takes place at a point where the
temperature change $(\partial T/\partial x)$ is maximal.

$$\text{div } v = \frac{D_o \, eZ^* \, \rho \, j \, Q}{k^2 T^3} \, e^{-\frac{Q}{kT}} \frac{\partial T}{\partial x}$$

Voids and Hillocks

In Fig. 4, several scanning electron micrographs of an aluminum film which was subjected to a current density of 5×10^5 A/cm^2 are shown. Voids near the cathode and hillocks near the anode can be clearly seen.
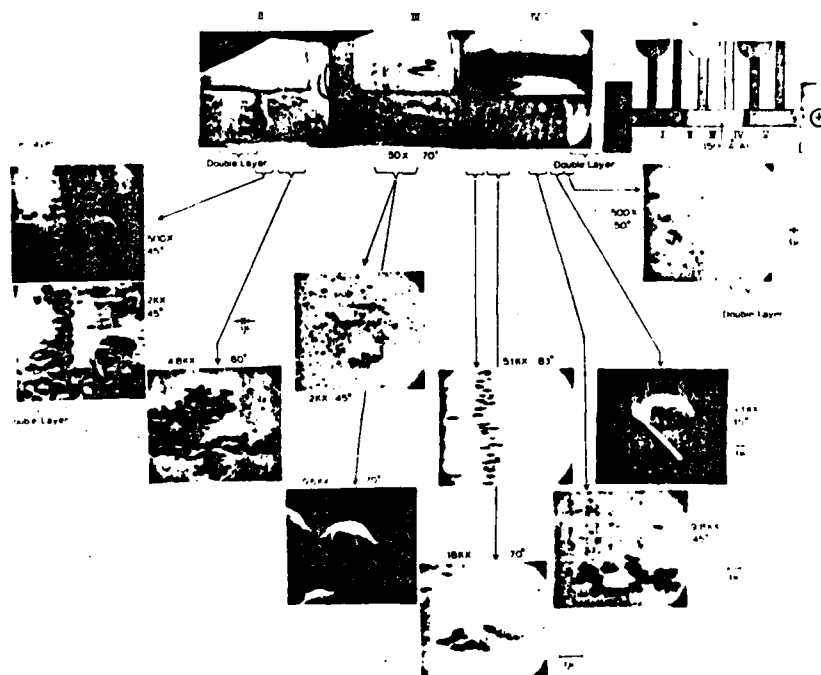

Fig. 4. Scanning electron micrographs of various areas of an aluminum thin film subjected to 5×10^5 A/cm^2. The shaded area represents the thicker electrode which overlaps partially the actual thin film (double layer). Note the whisker which grew out of one of the hillocks. From Ref. 15.

The importance of grain boundary migration is observed in Fig. 5 in which an aluminum film is shown after various times of current stressing.[16] After a particular grain boundary (2) has turned into a void by grain boundary grooving, no further ion transport takes place via that path. As a consequence, the hillock (3)
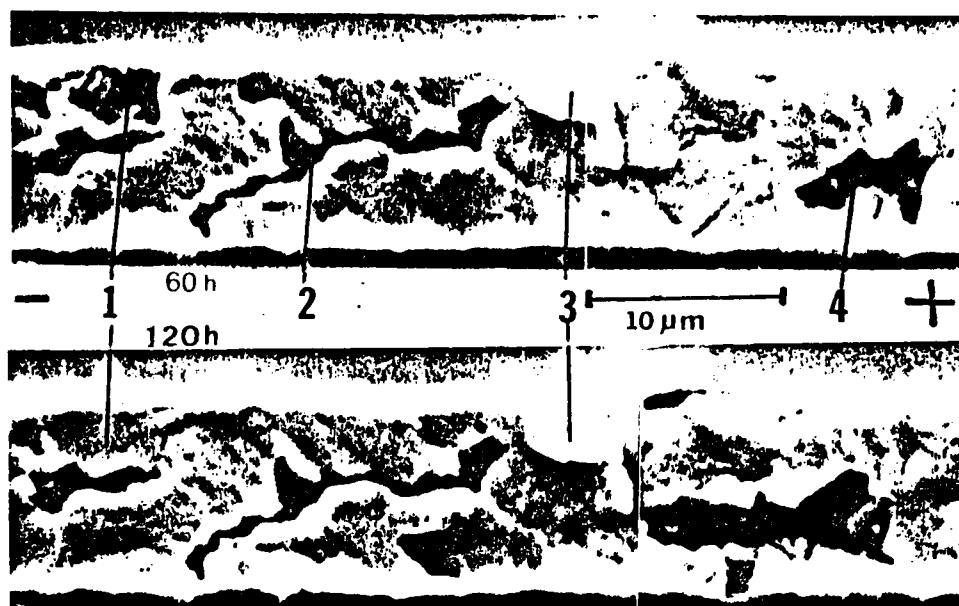
Fig. 5.   Scanning electron micrographs of an aluminum
          thin film after two times of current stressing.
          Explanation see text.   From Ref. 16.

which received material through the grain boundary does
not grow any further and the void from which the mate-
rial was taken (1) fills up. However, another void
further down towards the anode (4), still grows.

     The hillocks of various metals often have peculiar
shapes, for example the growths on indium films appear
almost spherical (Fig. 6), whereas the growths on tin
films look as if they had been extruded out of the sur-
face (Fig. 7).

Local Gradients
     Besides gross temperature gradients along the
stripe, also local temperature gradients, caused by
non-adherence of the film on the substrate ("hot
spots") or thickness variations, take place. As a con-
sequence, hillocks and voids can be observed to a
lesser extent virtually anywhere on the films (Fig. 3).
Even voids and hillocks next to each other are not un-
common. The failure time and the place where voids are
first formed are therefore basically of statistical
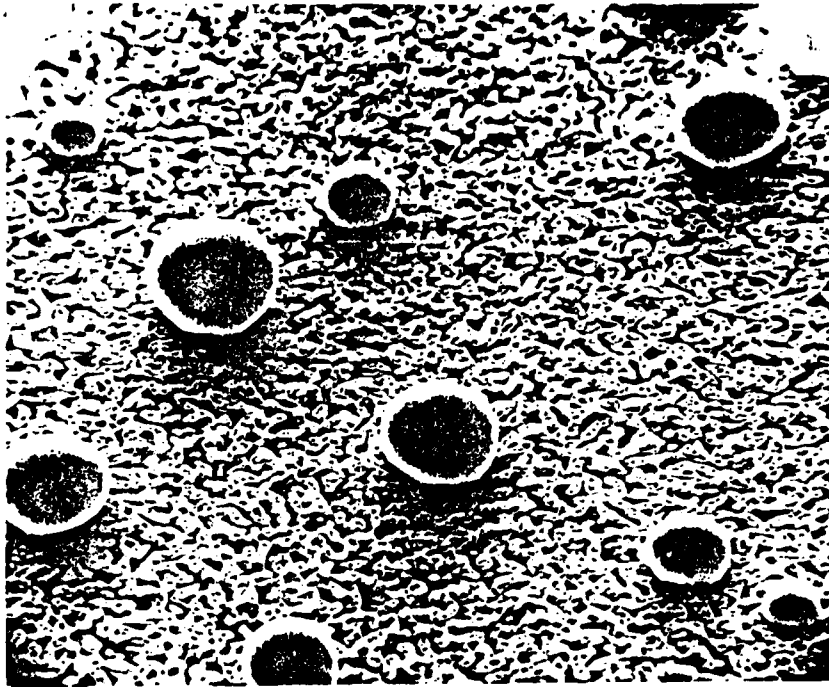nature.

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Fig. 6.   Scanning electron micrograph of growth on an
indium thin film after dc current stressing
($j=1.2\times10^5$ A/cm^2).   From Ref. 24.

Fig. 7.   Scanning electron micrograph of a growth on a
tin thin film after dc current stressing ($j=1.2\times10^5$ A/cm^2).

Divergencies Due to Grain Size Gradients

As mentioned already, gradients in grain size and other structural parameters can also lead to void and hillock formation. This is demonstrated in Fig. 8, de-
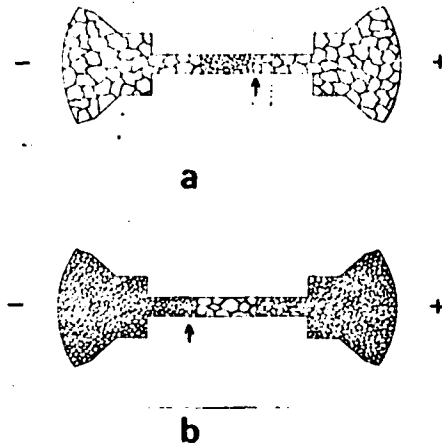

a

b

Fig. 8. Thin films with grain size gradient. The arrows mark the positions where hillocks are observed in aluminum films.

picting two thin film conductors consisting of areas of various grain sizes. (The two parts can be prepared in subsequent vapor depositions using different substrate temperatures.) Since thin film electrotransport is dominated by ion migration in the grain boundaries (see Fig. 1), one would correctly expect that a junction between a fine and a coarse grained area would cause void or hillock formation. In a unit volume encompassing such a function (Fig. 8a), less ions leave the coarse grained area via grain boundaries than are entering it. As a consequence, material accumulation (hillock) is observed at the separation between fine and large grained areas. The places where hillocks are observed are marked in Fig. 8 by arrows for the case where the ion transport is directed towards the anode.

Local grain size gradients lead likewise to hillock (and void) formation. It can be observed, for example, that a grain boundary triple point, i.e., a junction of three grain boundaries having two paths in and one path out, leads to a local accumulation of material. Also, a large angle between grain boundary and direction of electron flow can lead to flux divergencies.[17]

A reduction of the number of grain boundaries as achieved in coarse grained films or, as an extreme, in a single crystal, will lead to a diminishing ion flow. It has been shown[18] that a single-crystal aluminum thin film conductor subjected to a current density of 2 x 10^6 A/cm^2 did not reveal any signs of electromigration-induced failure even after 15,000 hours of current stressing.

Solute Effects on Electromigration

The effect of additions of small amounts of impurities to thin film metallizations has drawn considerable interest since it was found that additions of copper were effective in reducing electromigration damage in aluminum films.[19] The solubility of copper in aluminum is very small (about 0.05 at.% at $250°C^{20}$). Thus, copper is likely to segregate at the grain boundaries, and block the aluminum migration in the grain boundaries to a certain extent. Progressing electrotransport eventually moves the copper along with the aluminum towards the anode until failure finally occurs.[21] Copper solutes move therefore in the same direction as the aluminum solvent. This has been convincingly demonstrated by Howard and Ross,[21] who vapor deposited a small copper dot onto the center of a pure aluminum conductor. (After the copper was allowed to diffuse into the aluminum by proper heat treatment, the excess copper was etched away.) The copper atoms segregated at the grain boundaries of the center portion of the aluminum strip and created a divergency in ion flux because the velocity of aluminum migration is reduced in the copper-doped region (Fig. 9). After dc current stressing, aluminum hillocks were observed at the cathode side of the copper dot, i.e., at the opposite place to electrotransport in pure aluminum films. Progressing electromigration moves the hillocks further into the copper-doped area so that the hillock movement represents a direct observation of the displacement of copper by the electron wind.

Similar blocking effects have been observed for magnesium[22] or nickel[23] additions to aluminum. Both solutes have like copper only a small solubility in aluminum.

Direction of Electrotransport in Thin Films

The direction of electrotransport in some metal films contains still interesting questions. There is general agreement that the ion movement in aluminum,
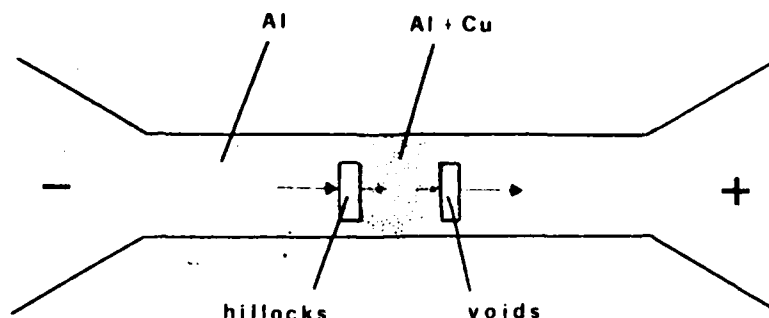
Fig. 9. Schematic representation of an aluminum thin film containing a copper-doped area at the center, during electrotransport.

indium, lead, tin or magnesium films is towards the anode, i.e., in the same direction as the "electron wind."[24] This has been substantiated by several experiments such as direct observations with the optical or transmission electron[25] microscopy, by using the scanning electron microscope[26] or the microprobe as well as by indirect techniques such as resistance measurements.[15,27] It was found unanimously that voids are formed at the cathode side of a thin film strip consisting of these metals, whereas hillocks were observed at the anode. Once the same techniques were applied for gold, silver or copper films deposited on microscope glass substrates, the voids were found on the anode and the hillocks on the cathode,[24,28,29] suggesting electrotransport against the electron flow. Likewise, the resistance increase in silver, copper and gold films is largest near the anode, i.e., at the place where voids are formed.[28] This behavior is not observed when transition metal underlayers (e.g., Cr, Ti-W, Ni-Fe, Ti, or Mo between the gold film and the substrate) are used.[30-36] In this case, the behavior of voids and hillocks are found to support electron wind arguments (i.e., voids segregate on the cathode and hillocks on the anode). Blech and Kinsbron[36] have, in addition, demonstrated that the edge of a gold film which was deposited onto a molybdenum layer moves towards the anode, leaving a free molybdenum surface on the cathode.

Considering the experimental evidence one has to look upon electrotransport in silver, copper and gold

films as a phenomenon where impurities stemming from
the substrate or the underlayer play an important role.
This suggestion is confirmed by studies where gold was
laid on a chromium underlayer.[35] Auger analysis showed
that chromium migrates due to heat treatment (annealing
or Joule heating of the stripe) through the gold
film[37,38] and is deposited as chromium oxide on the
free gold surface (Fig. 10). The preferred paths for
this chromium migration through gold is via grain
boundaries.
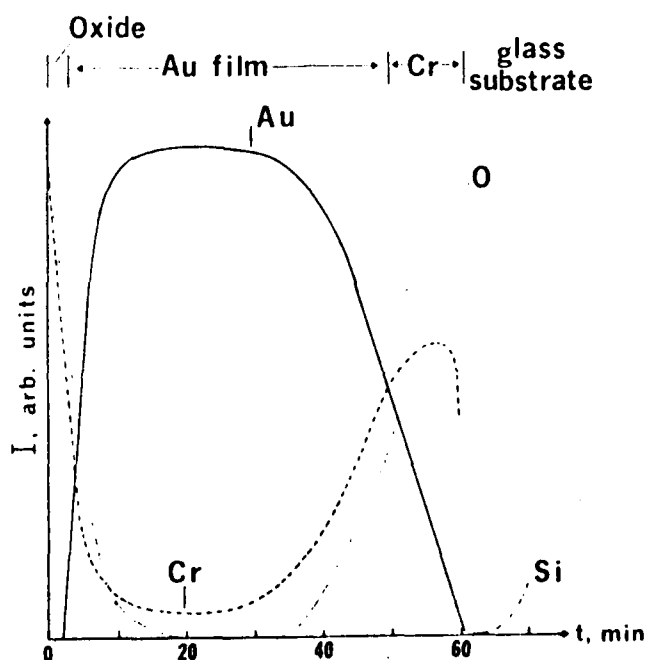
Fig. 10. Auger analysis of an annealed gold film vapor
deposited on a molybdenum underlayer. The
abscissa shows time of ion milling in minutes.
From. Ref. 35.

A possible way of explaining the void formation
near the cathode in gold films deposited on chromium
or other underlayers could be done along similar lines
as described in Fig. 9. One could argue that because
of the higher temperature at the center of a gold film,
the gold is doped here most with chromium causing a di-
vergence in ion flux. The void formation near the
cathode in gold on chromium films would support then

speculations suggesting ion transport against the electron motion. However, this argument could work also the other way around: we have found that gold films deposited on quartz glass slides or on regular microscope slides covered with a sputtered layer of SiO_2 show holes and hillocks to form like in pure aluminum, i.e., holes at the cathode. Further, we observed, using Auger analysis of gold films deposited on microscope glass, some sodium in and on the gold film which clearly stems from the sodium in the glass. Again, a higher sodium concentration in the center part of the film could cause a divergence in ion flux. Further investigations which are in progress will hopefully solve this interesting question.

Conclusions

This review intends to demonstrate that a strong interaction exists between thin metal films and the substrate on which these films are deposited. The substrate has a decisive influence on the electromigration behavior because of the rapid diffusion of foreign atoms contained in the substrate through the grain boundaries of the films. Therefore, thin film technology has to take increasingly into consideration the nature of the substrate.

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The effect of minor constituents on the electrotransport-induced failure site in thin gold films

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The presence of small amounts of some solute elements in gold thin films causes a reversal of the failure site during an electrotransport experiment. The effectiveness of a solute element in accomplishing this reversal is not correlated with melting point, solubility, or atomic radius. However, solute elements that produced a reversal in gold films (sodium, indium, barium) all have low ionization energies. Auger electron spectroscopy revealed that sodium from a microscope glass substrate penetrates through a gold film to its free surface during current stressing or during furnace annealing.

Failure of microelectronic devices often results from the formation of voids in the -film metallizations that develop as mass is transported fro. one region of the film to another under the influence of an electric current and a temperature (or ac) gradient. This electrotransport phenomenon has been described in terms of momentum transfer between the moving electrons and the metal ions in the film. As a result of this "electron wind" and temperature gradient voids are expected to develop near the cathode with a corresponding buildup of matter at the anode. In gold, silver, and copper films, however, voids have been found to form near the anode.[1–3] Some investigators[4–11] have contested these observations. In this letter, we present some experimental evidence that the site of electromigration-induced void formation in gold films is influenced by minor constituents.

Stripes of gold (purity 99.99%), 1000 Å thick, were vapor deposited in high vacuum (10^{-5} Torr) as described elsewhere[3] on quartz glass substrates. When subjected to a dc current of a current density of characteristically 5×10^5 A/cm², void formation and failure due to electrotransport were observed to occur in these films near the cathode side of the stripe.

In a second set of experiments, approximately 100 Å of an additional metal was vapor deposited (or, in one case, sputtered) either on top of the gold film or sandwiched between the quartz glass substrate and the gold film, and subsequently annealed in helium atmosphere for diffusion at 350 °C for 2 h. The failure site after current stressing was investigated by means of optical microscopy as well as microprobe analysis. It was found that among the solutes which were selected as second constituents, sodium, indium, and barium caused the gold film to fail near the anode, whereas gallium, lead, zinc, chromium, and molybdenum did not cause a change of the failure site compared to pure gold (see Table I).[12–14]

The solutes investigated can be divided into those with low and others with relatively high melting points. Among the low-melting solutes, sodium and indium reverse the failure site in gold, whereas gallium does not (Table I). Barium, however, which has a melting point of 714 °C, reverses the failure site of gold films. It is concluded that the location of the failure site is not simply related to the melting point of the solute added.

The solutes may also be divided into those which are readily soluble in gold and others which are not (Table I). Among the "reversing solutes" is one which has negligible solubility in gold (sodium) and one which has a solubility of approximately 8% at room temperature (indium). Likewise, among the solutes which do not reverse the position of void formation are species with practically no solubility in gold and some with high solubility. It is therefore concluded that solutes which show reversing of the failure site cannot be selected by the criterion of solubility. Similarly, as shown in Table I, the atomic radius of the solute does not provide a basis for the selection of solutes which cause reversal.

Inspection of Table I shows, however, that solutes which reverse the failure site in gold films all have an ionization energy which is smaller than 6 eV. Since electrotransport in thin gold films predominantly uses the grain boundaries as pathways,[15] this result could mean that the electronic structure in the grain boundaries is altered by the presence of the solute.

TABLE I. Summary of solute effects on the failure site of thin gold films deposited on quartz glass[a].

Solute	Failure site	Solubility of solute in gold at room temp. (see Ref. 13) (at. %)	Melting point of solute (see Ref. 13) (°C)	Ionization energy of solute (see Ref. 15) (eV)	Atom radius (see Ref. 14) (Å)
No solute	Cathode	···	···	···	···
Na[b]	Anode	0	97.8	5.14	1.9
In	Anode	≅ 8	156.2	5.79	1.66
Ba	Anode	?	714	5.21	2.22
Ga	Cathode	≅ 10	29.8	6	1.41
Pb	Cathode	0	327.4	7.42	1.75
Zn	Cathode	≈ 14	419.5	9.39	1.38
Cr[c]	Cathode	≅ 20	1875	6.77	1.27
Mo[c]	Cathode	≈ 0.5	2610	7.1	1.39

[a] All electrotransport experiments reported in this table were carried out in air at ambient temperature.
[b] The sodium was vapor deposited onto the gold films. The substrate was quartz glass. In other experiments, microscope glass slides were used as substrates. Here, the sodium diffused from the glass slide into the gold film during Joule heating.
[c] Chromium and molybdenum were sandwiched between substrate and gold film. Chromium was vapor deposited, molybdenum was sputtered.

TABLE II. Summary of Auger electron spectroscopy of Au films.

Substrate	Deposited solute	Heat treatment	Results	Failure site
Quartz substrate				
Quartz	Na vapor deposited	1.5 h at 350°C plus Joule heating	Na on surface and in film; most Na on surface and near center	+
Glass substrate				
Glass	None	No heat treatment	Virtually no Na on surface; none in film	Not run
Glass	None	72 h at 100°C in furnace	Na on surface; virtually none in Au film	Not run
Glass	None	100 h Joule heating at approx. 150°C	Na on surface	+
Glass	None	5 h at 300°C in furnace	Most Na near surface; virtually none in film	Not run
Solutes other than sodium				
Quartz	In vapor deposited	2 h at 350°C plus Joule heating	Only small amounts of In; no Na	+
Glass	Cr vapor deposited between Au and glass	Joule heating	Cr and traces of Na in Au film	−
Glass	Mo sputtered between Au and glass	24 h at 400°C	No Mo in Au but traces of Na on surface	−

Auger electron spectroscopy of thin gold films. Auger electron spectroscopy was employed to investigate the possible presence of various solute elements in and on the gold films used in this study. The Auger analysis was taken at the surface, and, in most cases, again after some gold had been removed by ion milling. The results are summarized in Table II.

Quartz glass substrates. Thin gold films were vapor deposited on quartz glass substrates and subsequently annealed in vacuum up to 75 h at 400°C. Auger analysis did not show any traces of second constituents in these films. However, when sodium was sandwiched between the quartz glass substrate and gold film and then annealed as above, a strong sodium peak appeared.

Glass substrates. Microscope glass (soda-lime-silicate glass) contains up to 10% sodium. If gold films are deposited on this type of glass and heated either in the furnance or during an electrotransport experiment by Joule heating, some of the sodium diffuses through the gold and appears on the free surface, Fig. 1. (This observation sheds some new light on the controversy of the failure site in gold films. When the gold film is deposited on soda-lime-silicate glass, the sodium which diffuses into the gold during Joule heating causes the reversal.) No sodium was detected at the free surface when a gold film on a microscope glass substrate was not subjected to any heat treatment (Table II).

Other solutes: indium. The solubility of indium in gold is approximately 8% at room temperature.[13] Indium was sandwiched between quartz substrates and gold films. After current stressing, indium could be found not only on the surface of the gold films but also after ion milling, i.e., in the films. The amount of indium found in either position was small; nevertheless, the indium that was present in the gold film caused a reversal of the failure site.

Chromium. The solubility of chromium in gold is substantial (approximately 20% at room temperature). Chromium was vapor deposited on a glass substrate and gold was deposited on the chromium. During Joule heating, the chromium diffused through the gold onto the free gold surface and segregated there in significant quantity.[1] (Along with the chromium, traces of sodium were detected with Auger spectroscopy.) Despite the presence of a substantial amount of chromium, this gold film did not show a reversal.

Molybdenum. Molybdenum was sputtered on a glass substrate and gold was vapor deposited on this intermediate layer. No molybdenum could be found in the gold film after electrotransport. This film did not show a failure site reversal.

Other solvents: Aluminum. Thin aluminum stripes are known to fail during an electrotransport experiment near the cathode, i.e., consistent with electron-wind arguments. The choice of the substrate does not change the failure site. The solubility of sodium in aluminum is practically nil. Nevertheless, as in the gold case
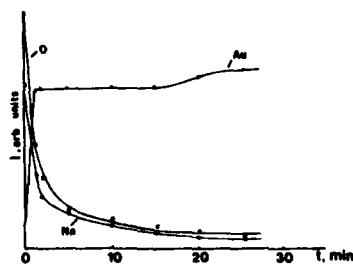

FIG. 1. Intensity of Auger peaks taken from a gold film which was deposited on a microscope glass slide. (The gold stripe was Joule heated before Auger graphs were taken.) The abscissa is the milling time which is proportional to the film depth (3 min ≈ 100 Å).

above, sodium was detected on the free surface of aluminum as well as in the film when aluminum was deposited onto microscope glass slides. The question of why sodium reverses the failure site in gold but not in aluminum remains unanswered.

In summary, these investigations have shown that among the solutes which were investigated, those constituents with low ionization energy cause a reversal of the failure site in thin gold films during an electrotransport experiment, while solutes with high ionization energies do not. In aluminum films, however, sodium did not cause a reversal of the failure site.

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THERMAL GROOVING, THERMOTRANSPORT AND ELECTROTRANSPORT IN DOPED AND UNDOPED THIN GOLD FILMS

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Thin gold films, undoped and doped with sodium or indium, were deposited onto quartz glass substrates. In order to separate the contributions of thermal grooving, thermomigration and electromigration to structural evolution and hole formation, series of samples were subjected to three treatments: (1) an isothermal anneal which produces only thermal grooving; (2) application of an a.c. which sets up temperature gradients and potentially produces thermal grooving and thermotransport; (3) application of a d.c. which potentially induces thermal grooving, thermotransport and electrotransport. Examination of the resulting samples with transmission optical, transmission electron and scanning electron microscopy revealed that (1) all three mechanisms may contribute significantly to hole formation in undoped films, (2) addition of indium stabilizes the as-deposited grain size, minimizes thermal grooving but promotes hole formation by the other two mechanisms and (3) addition of sodium greatly enhances grain boundary grooving, promotes early failure in a.c.-stressed samples and results in hole formation at the anode under a d.c. These results do not provide a rationalization of the reversal in the site of hole formation in d.c.-stressed sodium-doped gold films in comparison with the "normal" behavior exemplified in undoped gold films. Three possible mechanisms are suggested which might explain this observed reversal in failure site.

1. INTRODUCTION

The lifetime of thin film metallizations used in microelectronic devices is limited by the development of holes which form, grow and ultimately interrupt the current flow. Studies of this mode of circuit failure have centered primarily on the role of electrotransport in producing the mass flow that leads to hole formation. The present study is based on the recognition that other mechanisms may also contribute to matter transport under conditions normally encountered in service. Mechanisms included in these considerations are as follows.

(1) In *grain boundary grooving* the imbalance of surface tensions (acting where grain boundaries meet external surfaces) produces mass transport in response to the tendency to pull the grain boundary traces on the free surface toward the substrate.

A stripe annealed isothermally may develop a uniform distribution of holes as a result of grain boundary grooving; where there is a temperature variation, holes tend to form in hot regions where the rate of grooving is rapid.

(2) *Thermotransport* is a process in which a temperature gradient induces mass transport[1,2]. Depending on the material of the stripe, atoms may migrate to either the hot or the cold end. In the first case, holes develop in cold regions; in the second case they form where the stripe is hot.

(3) In *electrotransport* mass transport is induced by an applied electric field[1,2]. Positively charged metal ions tend to migrate towards the cathode under the *electrostatic force* induced by the electric field. In metallic conductors where the charge is carried by an intense flux of electrons, an effective *electron wind force* also acts on the metallic ions, pushing them in the direction of the electron flow toward the anode. In metals the electrostatic force is thought to be small in comparison with the electron wind force, so that in most cases it has been neglected[3]. Where the electron wind force dominates, flow is from the cathode toward the anode and holes would be expected to develop near the cathode.

(4) *Diffusion* is a mechanism in which components flow under the action of a concentration gradient. In stripes containing two or more elements, responses to the other driving forces may develop concentration gradients which then drive a diffusion process. The contribution that diffusion might make to hole formation is specific to each individual system.

Hole formation is best viewed as resulting from cooperation between these mechanisms.

In order to understand the origins of hole formation in a given thin film stripe, it is necessary to assess the contributions of each of these processes. The investigation described in this paper was designed to separate these contributions to hole formation in thin films of pure gold and gold films containing sodium or indium. This separation is accomplished by first examining the response of films when they are annealed isothermally, thus producing grain boundary grooving only. Subjecting films to an a.c. produces no net electrotransport but sets up temperature gradients that permit assessment of the importance of thermomigration. Finally, the application of a d.c. field to a film superimposes electromigration on thermomigration and grain boundary grooving.

2. EXPERIMENTAL PROCEDURE

Thin gold films (of purity 99.99%) with an average thickness of about 800 Å were vapor deposited at ambient temperature from molybdenum boats in a high vacuum (approximately 5×10^{18} Torr) onto clean dry quartz glass slides. The stripes were 1.74 mm wide and 16 mm long with wider electrode areas at the ends. The deposition rate was typically 5 Å s^{-1}. The thickness was measured during deposition using a thickness monitor and was spot checked after deposition with an interference microscope. The gold films were annealed in a tube furnace in a helium atmosphere. Samples were current stressed in either a vacuum (10^{-2} Torr) or ambient atmosphere with no noticeable difference in response. A constant-current power supply controlled the current applied in the thermotransport and electrotransport experiments. The current was increased slowly during the initial 30

min of each experiment. The sample holder was a ceramic block which had a small thermal conductivity.

3. EXPERIMENTAL RESULTS

3.1. Annealing of pure gold films

Figure 1 shows a composite of photomicrographs of unannealed and annealed gold films as observed using scanning electron microscopy (SEM), transmission electron microscopy (TEM) and transmission optical microscopy (TOM). The following observations are made.

(a) The mean grain size increases during moderate annealing (190 C for 30 days) from 0.2 μm (unannealed) to 0.5 μm (annealed) (see transmission electron micrographs).

(b) After the same moderate annealing (190 C for 30 days) fine "pinholes" approximately 1 μm in size are visible by transmitted light in an optical microscope.

(c) Grain boundary grooving, first evident after 1 h at 340 °C, is pronounced after 3 h at 340 C and leads eventually to hole formation at 560 C (see Fig. 1, top row).

(d) Annealing also causes some blistering or "bubbles" (Fig. 1, center and lower rows). These large bubbles (1–20 μm in size) are believed to be caused by gases trapped between the film and the substrate or by other adhesion problems. During annealing, these blisters may burst and create additional holes (Fig. 1, center, right). The micrograph on the lower right in Fig. 1 shows evidence for both kinds of openings, i.e. those caused by burst bubbles and those caused by grain boundary grooving. (Andrew[4] has also observed bubbles after annealing vapor-deposited gold films on NaCl substrates at 300 C. These bubbles were, however, only 50 Å in diameter and were believed by Andrew to be due to gas which was trapped within the thin film during deposition.)

In summary, Fig. 1 clearly demonstrates that the annealing of pure gold films may destroy the continuity of the film without the presence of a temperature gradient or an applied electric field.

3.2. Annealing of gold films doped with sodium

Gold films were doped with sodium by one of two methods; both showed equivalent results. In one procedure, gold was vapor deposited onto quartz glass substrates and the sodium was added by vapor deposition. For the other method, gold was vapor deposited onto soda-lime silica glass (microscope slides) which supplied sodium to the gold film during annealing. The solubility of sodium in gold is extremely small. Any sodium picked up by the gold from either the soda-lime glass or the vapor-deposited sodium film approximately 100 Å thick is thus believed to segregate near grain boundaries. The amount of doping is estimated to be less than 10^{-3} atom fraction.

The scanning electron micrographs of sodium-doped gold films shown in Fig. 2 are arranged as in Fig.1. The following observations are made.

(a) Annealing at 340 °C for 1 h produces grain boundary grooving, hole formation and whisker growth.

(b) Further annealing for 3 h at the same temperature yields an increase in hole size.
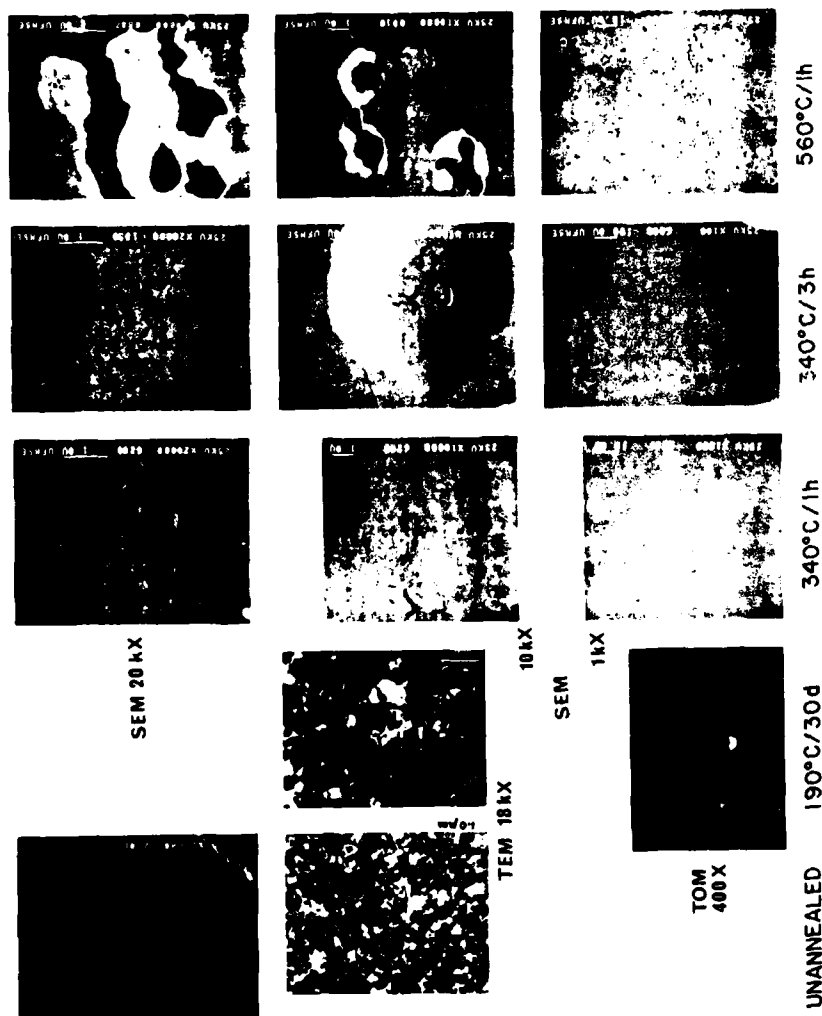
4

R. E. HUMMEL *et al.*

Fig. 1. Composite of transmission electron, scanning electron and transmission optical micrographs taken on annealed and as-deposited gold films. The annealing time and temperature increases from left to right. The magnification increases from bottom to top.

THERMAL GROOVING, THERMOTRANSPORT, ELECTROTRANSPORT IN Au FILMS   5

Fig. 2. Composite of scanning electron micrographs of sodium-doped gold films before and after annealing.

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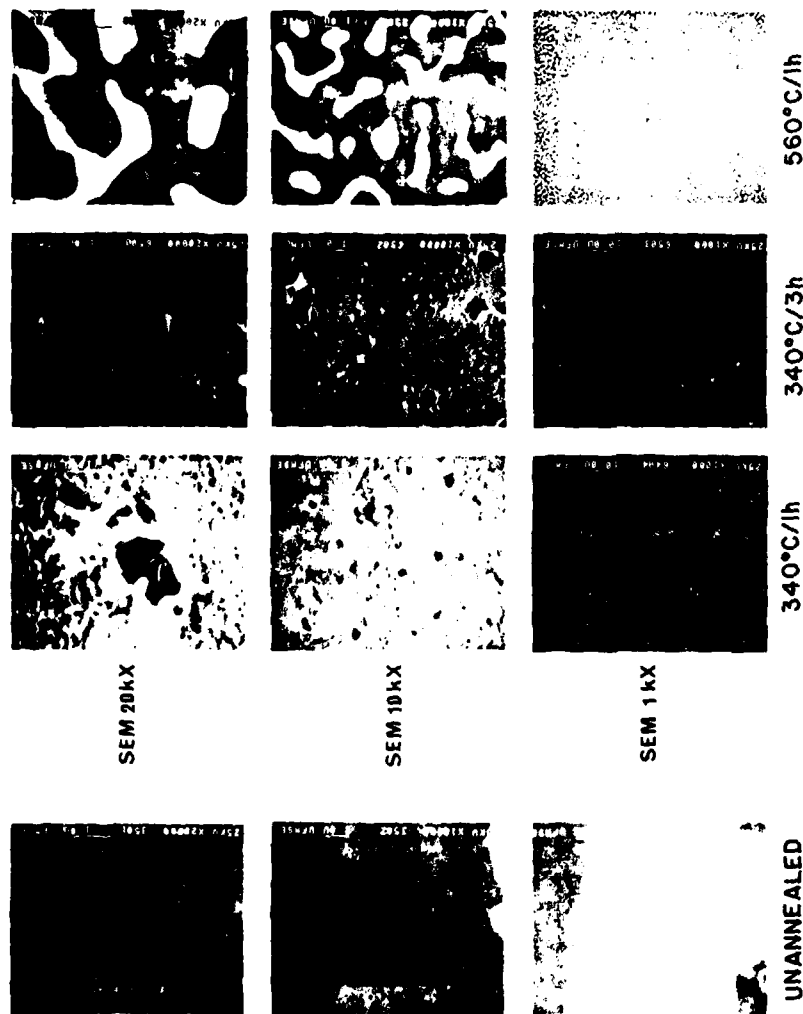
(c) When sodium-doped gold is annealed at 560 C, the film disintegrates into unconnected islands.

A comparison of Figs. 1 and 2 shows that grain boundary grooving and hole formation occur more rapidly in the sodium-doped gold films than in pure gold. However, the blisters observed in pure gold films are hardly visible in sodium-doped gold films. Further, at comparable stages of development of the pore structures, grain boundary grooving appears more pronounced in sodium-doped films than in pure gold films.

3.3. Annealing of gold films doped with indium

In these experiments an indium film 130 Å thick was sandwiched between a quartz glass substrate and a gold film 800 Å thick. (The vacuum was not broken between the two depositions.) The indium-to-gold ratio utilized here represents a Au 6wt.",In alloy, assuming that all the indium diffused into the gold. Visual inspection after annealing revealed, however, that some indium remained undissolved between the substrate and the gold film. Thus an accurate value for the amount of doping cannot be given at this time. (Usage of the word "doping" here is extended to include solute contents of a few per cent.)

The question arises whether or not an intermetallic phase had been formed during annealing. Two items should be considered which make this possibility unlikely.

(1) The solubility limit of indium in gold at the annealing temperatures used for most of these experiments is slightly above 6 wt.". Thus, even if all the indium had dissolved, the film would still be a solid solution.

(2) Layers of intermetallic compounds that may form during diffusion were not detected by electron diffraction.

As can be seen in Fig. 3 (which has the same format as Figs. 1 and 2), the unannealed film has some surface structure which appears to be related to the grain structure of the film. Annealing up to 560 C for 1 h does not noticeably increase the grain size nor does it produce additional grain boundary grooving. Thus indium doping seems to have a stabilizing effect on the grain boundary network. This is verified by comparing the transmission electron micrographs of Figs. 1 and 3: the indium-doped gold film possesses a fine grain size (approximately 0.05 μm in grain diameter) after annealing for 30 days at 190 C whereas the undoped film experienced grain growth to about 0.5 μm. Further, in the same film no pinholes are observed after this heat treatment, as evidenced by the transmission optical micrographs.

3.4. Thermotransport in gold films

The experimental results reported above show that annealing of undoped and sodium-doped gold films yields substantial hole formation, whereas holes are not observed in gold films doped with indium. Thus, in order to study ion movement due to a thermal gradient without possible disturbances by grain boundary grooving and similar effects, only indium-doped gold films may be expected to give unambiguous results.

An indium film 120 Å thick was sandwiched between a gold film (820 Å) and a quartz glass substrate. Thermotransport studies were undertaken by applying an

THERMAL GROOVING, THERMOTRANSPORT, ELECTROTRANSPORT IN Au FILMS   7

Fig. 3. Effect of annealing on indium-doped gold films. For details, see caption of Fig. 1.
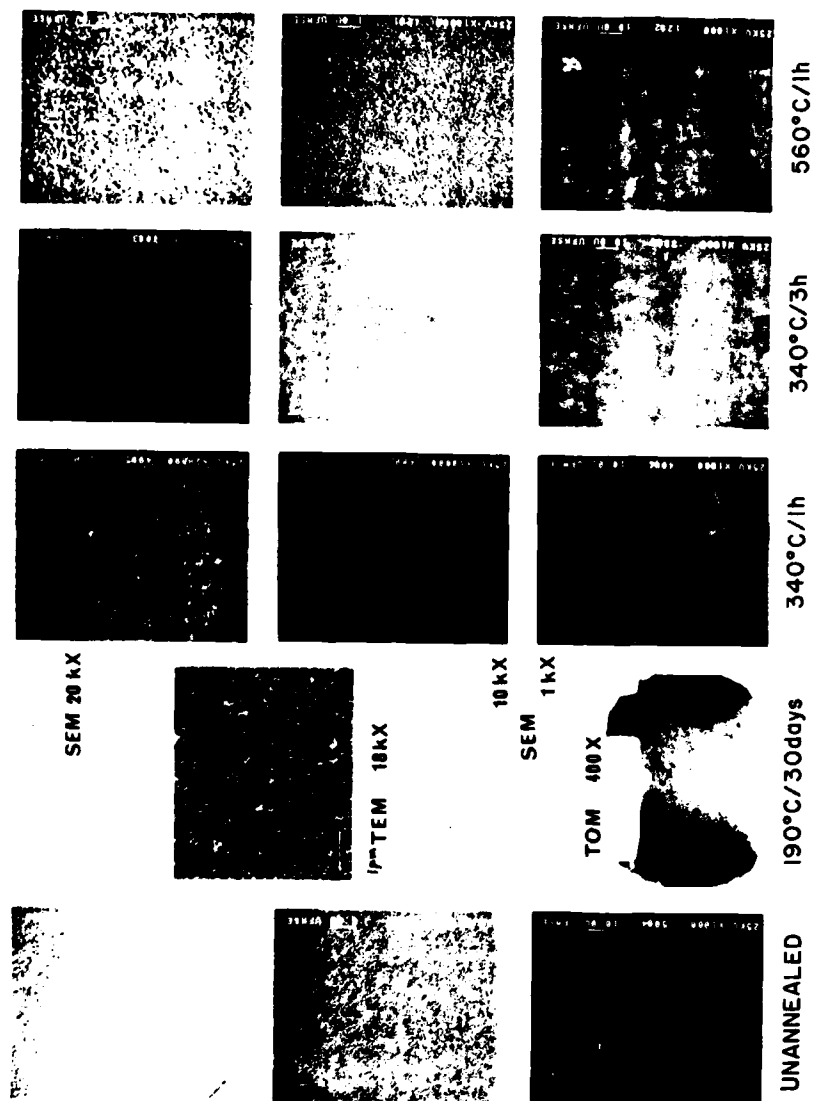
a.c. along the stripe. Figure 4 shows a transmission optical micrograph of such a film after 7 days of stressing with 1 A a.c. A peculiar elliptically shaped arrangement of thinned areas is evident. The center region, which is the hottest part of the stripe (approximately 350 C), has the highest optical density; thinning occurred at the colder parts (approximately 80 C). This pattern can be observed in three different areas: (a) along the length of the stripe; (b) across the width of the stripe; (c) on the colder potential probes. Application of the a.c. was maintained until failure of the thin film stripe near one of the electrodes had occurred. The location of the failure site is of a statistical nature; close inspection of Fig. 4 reveals that a similar failure could have occurred some time later near the other electrode where some connected holes are already visible. Figure 4 suggests that thermotransport in indium-doped gold films is directed from cold to hot regions. This result can be compared with findings on bulk gold where migration toward higher temperatures is also reported for this metal[5-7].

Figure 5 is a photomicrograph of a pure gold film 800 Å thick on a quartz glass substrate after 18 days of current stressing with 1 A a.c. Two processes are likely to occur simultaneously: (a) grain boundary grooving tending to form holes preferentially at the center of the stripe where the temperature is highest; (b) thermotransport of gold toward the hot central region. Thus we would expect thermotransport to reduce the rate of hole formation at the center. Further we expect these competing mechanisms to extend the lifetime of the stripe. This has indeed been observed. Failure had not yet occurred in a pure gold film after 18 days of a.c. stressing (Fig. 5). A similar film stressed under the same conditions finally developed several holes that became interconnected at the center and interrupted the current flow after 31 days. The observation that thinning and hole formation are spread more or less uniformly across the entire film width lends further support to the contention that thermotransport acts in conjunction with hole formation due to annealing.

Results of these experiments suggest that, in a sodium-doped gold film, failure due to a.c. stressing would occur earlier than in pure gold films and would be localized near the center, since sodium increases the rate of grain boundary grooving. This has indeed been observed. Figure 6 shows a sodium-doped gold film 800 Å thick after 65 min of stressing with 1 A a.c. The holes are almost entirely concentrated about the center, *i.e.* the hottest area. As a result the observed lifetime is considerably shorter for sodium-doped gold films than for undoped films.

3.5. Electrotransport in gold films

The results reported here supplement work published earlier[2]. In the light of the findings presented above, it might be expected that pure electrotransport in thin gold films can be observed only in very rare cases; grain boundary grooving and thermotransport in general also contribute to matter transport in d.c.-stressed thin films. Indium doping prevents thermal grooving but not thermotransport; therefore a superposition of electrotransport and thermotransport is expected in indium-doped gold films. Assuming that the electron wind effect dominates in electrotransport, thereby producing a mass flow from the cathode to the anode, and assuming further that thermotransport moves matter from the cold electrodes to the hot center, we might expect a loss of material near the cathode due to
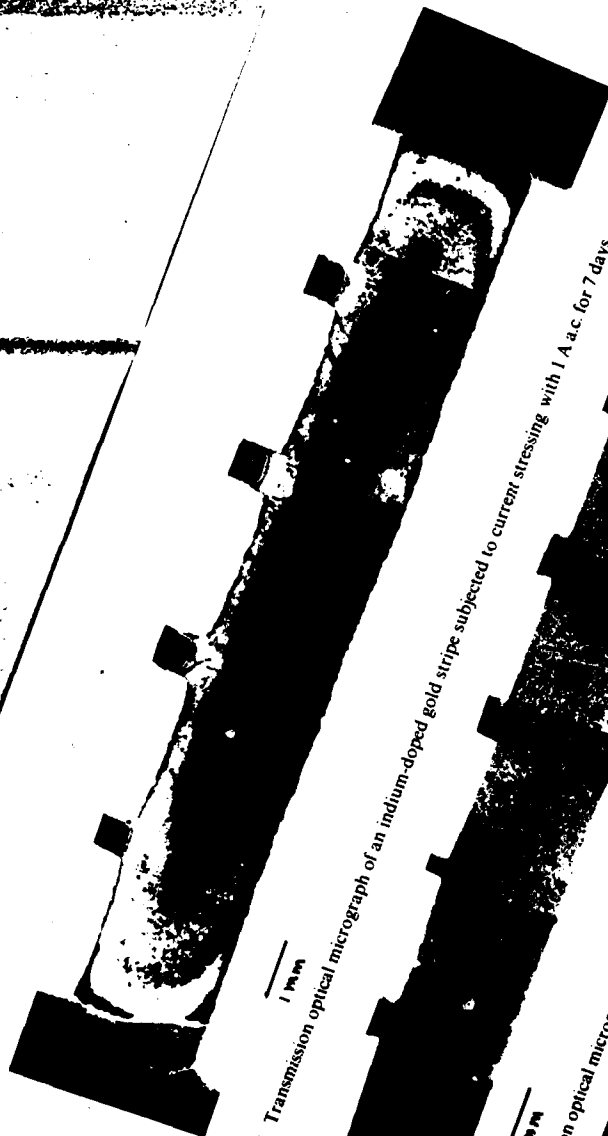
Fig. 4. Transmission optical micrograph of an indium-doped gold stripe subjected to current stressing with 1 A a.c. for 7 days.

Fig. 5. Transmission optical micrograph of an undoped gold film subjected to current stressing with 1 A a.c. for 18 days (current density, 6.25×10^5 A cm^{-2}).
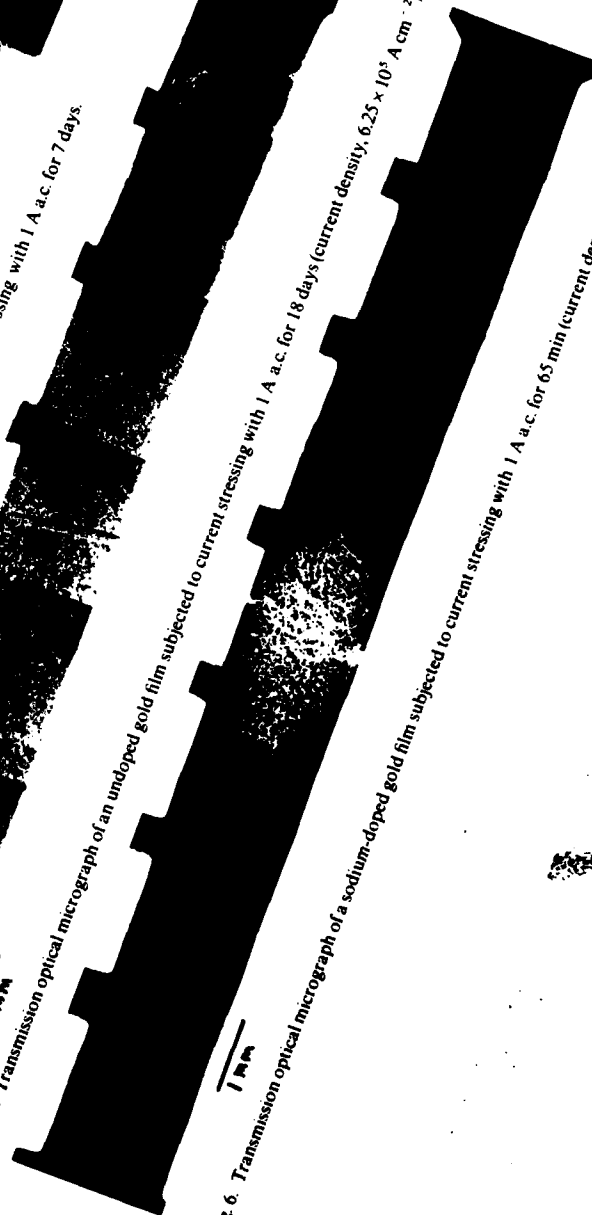
Fig. 6. Transmission optical micrograph of a sodium-doped gold film subjected to current stressing with 1 A a.c. for 65 min (current density, 6.2×10^5 A cm^{-2}).

10   R. E. HUMMEL *et al.*
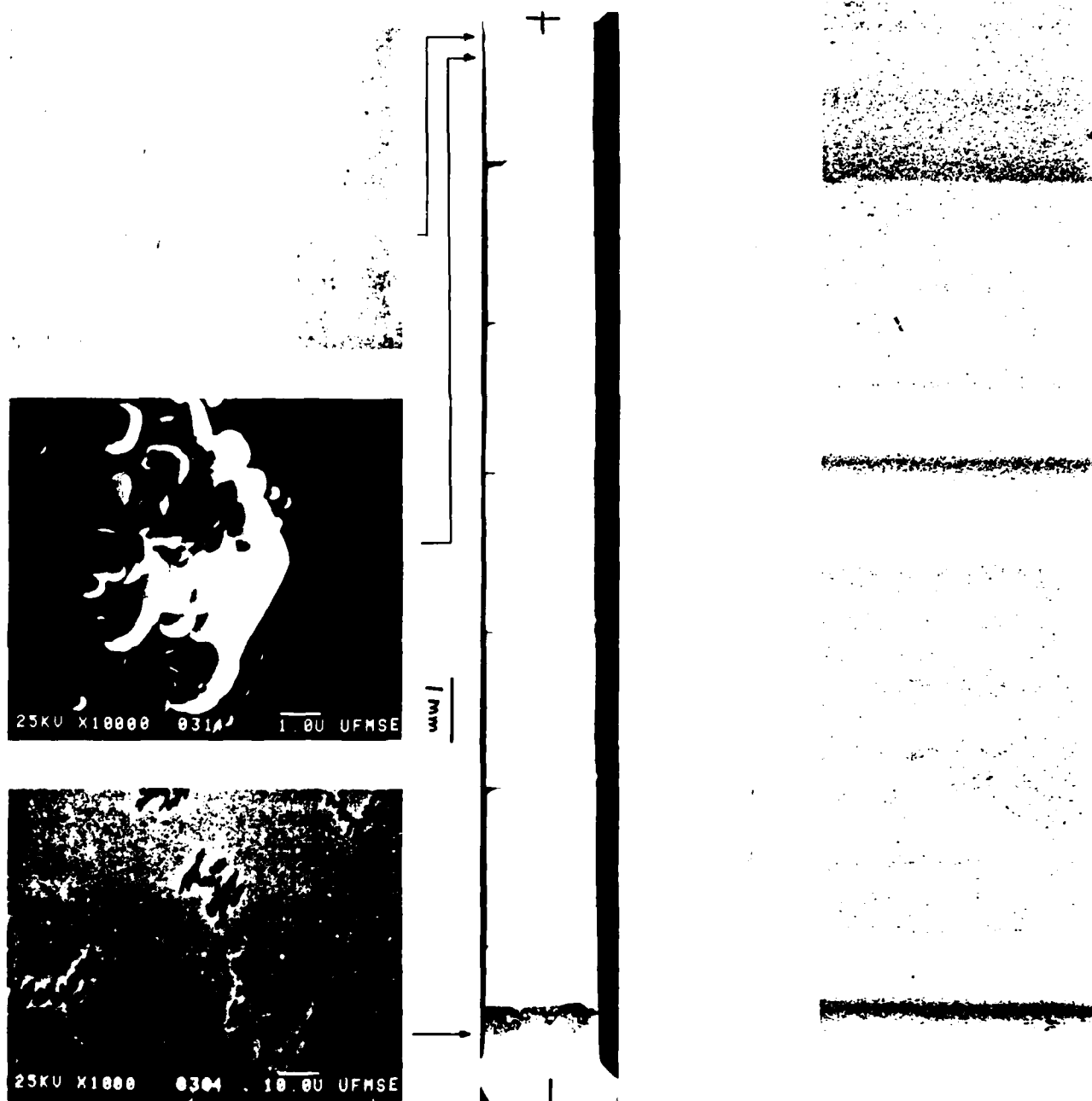

Fig. 7. Scanning electron micrographs of an indium-doped gold film subjected for 66.5 h to a d.c. of 0.84 A
(current density, 5.6 × 10⁻⁵ A cm⁻²). The optical micrograph was taken in reflection.

electrotransport as well as to thermotransport. An indium-doped gold film is therefore likely to fail near the cathode. The situation is probably more complex near the anode: we would expect a material loss there due to thermotransport, i.e. an ion flow toward the hot center which is counteracted fully or in part by the ion flow toward the anode due to electron wind effects. Figure 7 shows an indium-doped gold film subjected to stressing by a d.c. Void formation occurs indeed near the cathode[8].*. On the anode side, however, hillocks are observed with no visible hole formation. Furthermore, no holes can be detected around the center of the stripe (see the optical micrograph in Fig. 7). These observations suggest that in these films the contributions of thermotransport (and of thermal grooving) are small compared with that from electrotransport.

Finally, in pure gold films (800 Å gold on quartz glass) it is necessary to consider all three mechanisms, namely electrotransport, thermotransport and annealing effects. Our previous observations[8,9] as well as those of other investigators[10-15] generally show failure near the cathode, suggesting a predominance of electron wind effects. In some instances, however, grain boundary grooving, hole formation and even failure were observed to occur at the center of the stripe. In general this behavior was observed in samples in which the center of the stripe is very hot (approximately 350 C) and or the lifetime of the gold film is substantially longer owing to a larger grain size. Both of these factors favor failure by grain boundary grooving. Figure 8 shows a scanning electron micrograph of the center part of a pure gold film which was subjected to 1 A d.c. for 600 h. Failure had not yet occurred. Substantial grain boundary grooving is evident as expected. Similar grain boundary grooving has been observed by Kenrick[16] after annealing pure gold films on a chromium underlayer for 11 h at 300 C.

Fig. 8. Scanning electron micrograph of the central region of an undoped gold film that had been current stressed with 1 A d.c. for 600 h.

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* The preliminary observations reported in ref. 8 on the electrotransport-induced failure site of indium-doped gold films have not been substantiated.

12   R. E. HUMMEL *et al.*

4. DISCUSSION

The observations described above and summarized in Table 1 show that the failure mechanisms in thin film metallizations due to current flow are complex. An interpretation of the behavior of thin film stripes under current stressing should not be limited to electron wind arguments. Several failure mechanisms must be considered to act simultaneously. Eliminating one of them does not necessarily cure failure by another mechanism. For example, indium doping stabilizes the grain size in gold films and minimizes the role of grain boundary grooving. However, a small grain size is equivalent to a large number of grain boundaries which in turn provide the paths for mass flow by electrotransport and thermotransport at temperatures between about 250 and 400 C. Thus, although grain boundary grooving is eliminated, the lifetime of indium-doped films which were subjected to a d.c. is short. The longest lifetimes achieved in this work were for extremely pure gold films which lasted almost indefinitely at current densities of 5×10^5 A cm^{-2}. These films were finally brought to failure by increasing the current density and thus the temperature. Some of these films failed at the center, probably because of thermal grooving or related annealing effects.

Pure gold films deposited onto quartz glass substrates consistently fail near the cathode when stressed with a d.c.[8,9] This observation is in agreement with the assumption that electrotransport dominates over thermal grooving and thermotransport under the chosen conditions and that the electron wind force dominates in the electrotransport component of mass flow. However, gold films doped with sodium consistently fail near the anode[17-19]. This observation was a major impetus for undertaking the current investigation. Recognition that other mechanisms might contribute to mass flow offered a possibility for explaining this relocation of the failure site to the anode side of the stripe.

It has now been demonstrated that addition of sodium increases the rate of grain boundary grooving to such an extent that in a.c.-stressed samples this effect dominates over thermotransport. Thus, in d.c.-stressed sodium-doped samples, enhanced grain boundary grooving favors hole formation at the center of the stripe. Since thermotransport favors motion for gold from cold to hot regions, if this process is a factor, it would tend to form holes at both ends of the stripe. Neither of these effects favors the preferential development of holes at the anode as observed; accordingly, neither grain boundary grooving nor thermotransport provides an explanation for the observed reversal in failure site in sodium-doped films.

It is thus evident that the addition of sodium produces a fundamental change in the electrotransport component of mass flow under d.c. stressing. Three explanations for this observation have been proposed.

(a) The presence of sodium may reduce the magnitude of the electron wind force so that Coulomb forces may act to produce a net attraction of positive gold ions toward the cathode, thus creating holes near the anode. Since Coulomb forces are generally smaller than electron wind forces in pure gold, it would thus be expected that the rate of mass transport would be significantly reduced because of sodium doping in comparison with that which occurs in pure gold films. The addition of sodium would thus increase the lifetime of the film. However, it has been observed that sodium additions reduce the lifetime of the stripe.

TABLE I
SUMMARY OF RESULTS OBTAINED ON PURE AND DOPED GOLD FILMS

	Pure gold	*Gold doped with sodium*	*Gold doped with indium*
Annealing at 190–560 °C	Grain growth, grain boundary grooving, hole formation and blistering	Grain boundary grooving and pore structure are more pronounced than in pure gold films	Indium has a stabilizing effect on the grain boundary network. No hole formation
Thermotransport (1 A a.c.; temperature at center of stripe was about 350 °C)	(1) Mass transport from cold to hot part of the film (thermotransport) (2) Hole formation due to annealing, particularly at hot part. This hole formation at the hot part is partially counteracted by thermotransport	Same as for pure gold, but hole formation due to annealing is stronger. The result is fast failure near center of stripe	Mass transport from cold to hot part of film. Hole formation on cold parts of stripe. No hole formation due to annealing because of stabilizing effect of indium
Electrotransport (1 A d.c.; temperature at center of stripe about 350 °C)	*Cathode*: loss of gold due to electrotransport *and* thermotransport *Center*: hole formation due to annealing *Anode*: (1) accumulation of gold due to electrotransport; (2) loss of gold due to thermotransport	Observed void formation near anode (reversal of failure site) cannot be explained by grain boundary grooving or thermotransport	*Cathode*: loss of gold due to electrotransport *and* thermotransport *Anode*: (1) accumulation of gold due to electrotransport; (2) loss of gold due to thermotransport

14   R. E. HUMMEL *et al.*

(b) Using Auger analysis it has been shown that sodium diffuses through a 1000 Å gold film at 100 C in 5 min [8]. It is suggested that these highly mobile sodium ions might be attracted to the cathode by the Coulomb force; in the process they may exchange momentum with gold atoms in the grain boundaries, creating a "sodium wind" which counteracts and may even overpower the electron wind.

(c) Sodium may change the electronic structure in the grain boundaries so that a hole wind operates rather than an electron wind. This hole wind then produces a net flow of gold toward the cathode.

The experiments reported in this paper demonstrate that the effect exerted by the addition of sodium operates in the electrotransport component of flow. However, none of the current observations provides unambiguous support for any of the mechanisms that have been suggested to explain the formation of holes at the anode in sodium-doped gold films.

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"The Role of Sodium in the Electrotransport Behavior in Thin Gold Films"

The phenomenon of electromigration in thin films has long been of technical importance to the semiconductor industry. While its physical appearance has been well catalogued, the mechanisms by which electromigration operates have been less easily understood. Since electron flow is from cathode to anode, this electron flow—in thin films of very small cross section—causes a transport of matrix atoms in the same direction. This occurs since the electrons can transfer their momentum through collisions with these matrix atoms. After some amount of time, this momentum transfer can be noted in stripe failure due to material depletion near the cathode (often an actual open circuit) and material buildup near the anode (often seen as surface hillocks). In all film compositions to date this directionality in electrotransport failure is easily predictable. In contrast, the direction of failure is reversed for Au films containing Na. This reversal occurs with material depletion near the anode and material accumulation near the cathode. This material transport can take on a variety of forms, which shall be evident by the end of this report.

The direct detection of minute levels of sodium in thin gold films is by no means a trivial experiment. The introduction of Na into such films and its eventual migration due to various gradients in temperature, composition and an electric field cannot be easily tracked. Not only is the sodium concentration extremely low when compared to the matrix material (our work deals with Na levels of 50 ppm to 4.83 at.%), but its reactive nature also makes identification difficult. Almost any environment during analysis can affect the out-diffusion and/or oxidation of elemental Na, making its identification impossible. For example, the required specimen preparation for wet

chemical analysis as well as transmission electron microscopy (TEM) eliminates all traces of sodium due to the required solution pretreatment of the sample. Sodium's low atomic number of 11 makes its identification below the resolution limits of most electron optics instrumentation, such as the EDS (energy dispersive spectrometer) microprobe,[1] scanning electron microscopy (SEM) in the backscattered mode, etc. While secondary ion mass spectroscopy (SIMS) and Rutherford backscattering (RBS) techniques have the potential for Na detection, we did not have these analytic techniques at our disposal during the course of this study.

Since earlier work conducted in this laboratory has indicated that sodium (and to date, only sodium) produces a dramatic effect upon the electromigration of gold, it became apparent that sodium's effect needed to be studied, albiet indirectly. For reasons to be noted later in this report, we have studied the effect of sodium in gold films by introducing the impurity through various doping procedures. Previous work has led to the reproducible introduction of Na through the use of soda silicate (micro-scope) glass slides onto which the gold films are vapor-deposited. After deposition, as the films are powered, the Joule heating at the film-substrate interface is sufficient to cause the Na to leave the glass substrate and to enter the Au by way of the film's grain boundaries. This source of sodium is never depleted, since grain boundary volumes in a typical film are roughly 9 $\,$ x 10^{-9} cm^3 and the soda silicate substrates contain approximately 10% Na_2O or . $\,$ 10^7 times the amount of Na needed to saturate the Au grain boundaries. At the temperatures of 250-350°C which are experienced by the film-substrate interface during circuit powering, the Na, occupying rather loosely-bound interstitial sites in the glass, easily migrates through the glassy SiO_2 network to the film.

Questions arise, however, in the use of this doping procedure. First, the use of a semi-infinite Na source does not allow for varying the level of doping in the films. Second, all soda-silicate glasses contain other impurities, without which glass stability would be unapproachable. (For example, B_2O_3 and Al_2O_3 physically strengthen the glass and As and SB are often added to reduce bubble formation during glass manufacture.) Do these impurities act to retard or enhance Na's effect in Au? Finally, since sodium acts as a network modifier in silicate glasses, it is generally assumed that Na_2O rather than elemental Na resides in the glass. Does sodium or sodium oxide actually enter the Au film and cause reversal in gold electromigration? To eliminate these queries, a number of doping techniques were recently examined. These included:

    a) Vacuum furnace doping of Na

    b) Soda-silicate (microscope) slide doping

    c) Doping with fused glass slides (having a different Na concentration than (b))

    d) Ion bombardment of Na

Each technique will be specifically described in this report.

All Au films in this study ranged in thickness from 800 Å to 1000 Å with a common configuration that gave rise to a surface area of .25 cm^2. The procedure for the vapor deposition of the pure Au films has been described elsewhere.[2] After the appropriate doping procedure, all samples were tested and examined in the identical manner. Each sample was powered in a standard DC circuit, generally to failure. Individual experimental parameters will be listed with the appropriate samples.

The samples were then coated with a 15 Å vapor-deposited Al film for SEM examination. The coating prohibited electron beam charging due to the insulating nature of the glass substrate. All micrographs contained in this report were taken in the secondary mode with typical operating conditions including a 25 keV accelerating voltage, an emission current of 100 to 120 microamps, and a working distance of 15 mm. The SEM study allowed for close examination of the surface of each film. Such a surface study can easily follow the progress of electromigration in thin films.

Furnace Doping of Sodium

Pure Au films, deposited on high purity quartz slides were placed in the cooler, end portion of an evacuated tube furnace. Na was then placed in a molybdenum crucible which was also positioned in the furnace, but located in the center of the tube. The tube was then heated at 270°C for 70 minutes. During this heating segment, a vapor pressure differential is set up between the two regions of the furnace tube, causing evaporation from the Na source and condensation of the Na onto the films. The films were then cooled in the furnace, removed, and placed in the electrotransport circuit for powering.

Figure 1 depicts a schematic diagram of a typical sample subjected to this doping procedure after experiencing a current density of 7.34×10^5 amps/cm^2 for 384.5 hours.

The general appearance of the diagram will be maintained during all further sample descriptions. Boxes along the Au stripe indicate the areas where the appropriate SEM micrographs were taken. As the electrode labels indicate, electron flow occurs from right to left, i.e., from cathode to
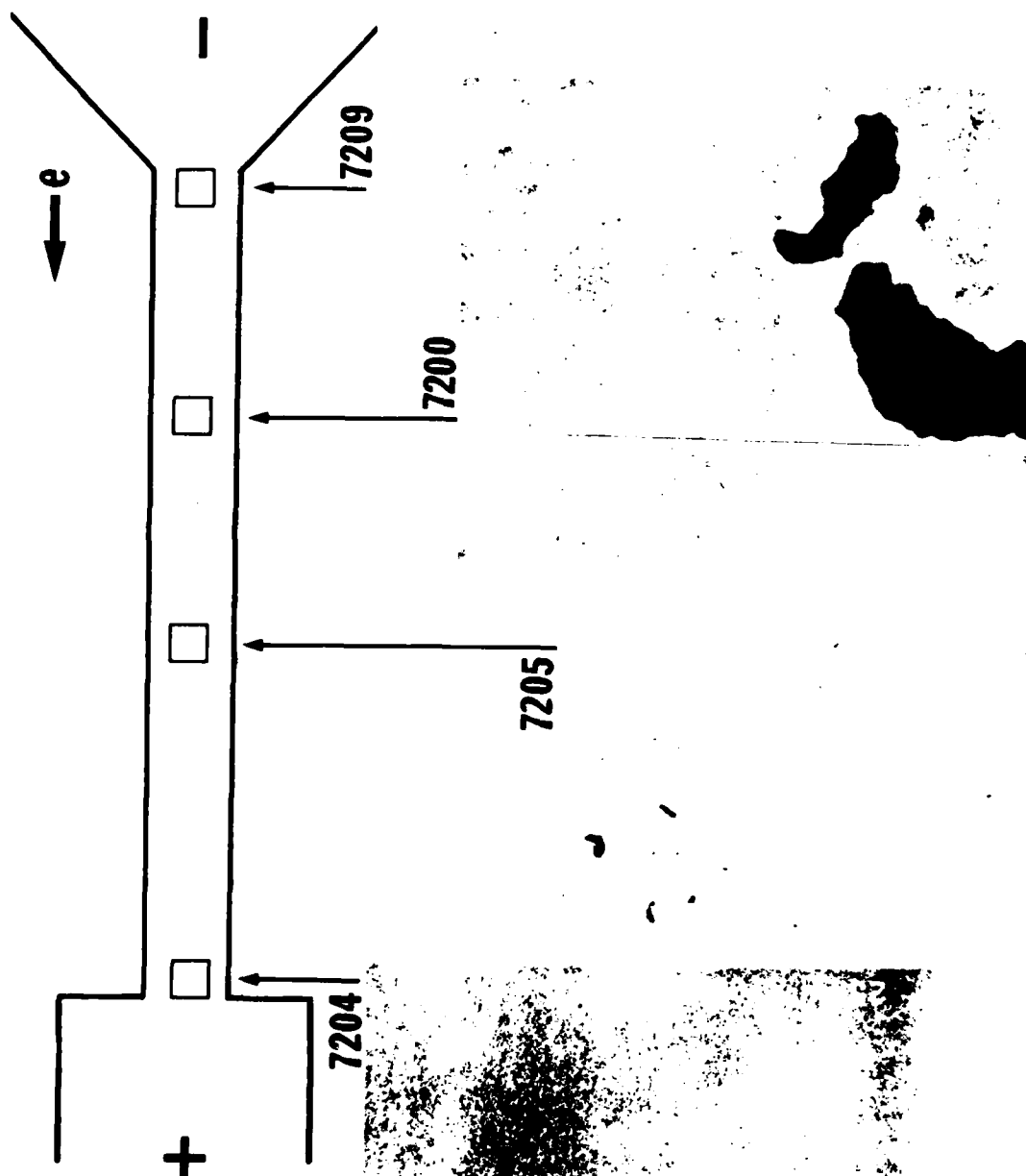
Figure 1. Sample GAu8A, gold film 815 Å thick on quartz substrate, Na-doped in furnace (280°C/70 min.), DC stressed at $J = 7.34 \times 10^5$ amps/cm2 on ceramic substrate holder for 384.5 hours. Current stressing terminated prior to failure.

anode. Signs of failure in Na-doped gold would consist of material depletion at the anode (+ electrode) and material accumulation near the cathode (- electrode).

Na Doping Through the Use of Soda-Silicate Glass Slides

As was mentioned arlier in this report, soda-silicate glass contains a sufficient amount of Na_2O (see Figure 5) to act as an infinite source of Na for the gold films, because the temperatures reached during resistive heating are adequate to cause diffusion of Na into the Au film.

Figure 2 schematically depicts GAu8E, an 815 Å film deposited on standard microscope slide. The sample experienced a current density of 5.72×10^5 amps/cm^2 for 237 hours. As SEM micrograph #7105 indicates, the anode surface is very clean with some small pinholes dispersed uniformly throughout the area. The central portion of the stripe becomes increasingly rough and shows small hillocks (SEM micrograph #7101). Lack of adhesion, as exemplified by the "bubble" in this micrograph, is also apparent. Bubble density and size become increasingly dramatic near the negative electrode. No holes are present, while some minor hillock formation which is associated with the roughened circular regions can be noted. One postulate used to describe this long-range asymptotic behavior is that a Na-containing compound collects at the Au-glass interface and is electrostatically attracted to the cathode where de-adhesion eventually occurs due to coalescence. Again, the surface features indicate an effect that opposes the direction of electron flow.

The sample depicted in Figure 3 consisted of 795 Å of Au which was also deposited on a soda-silicate glass. The sample failed after powering for 474 hours at 6.11×10^5 amps/cm^2. Some surface roughening is visible near the anode area (SEM micrograph #8056). Hole formation in this region is
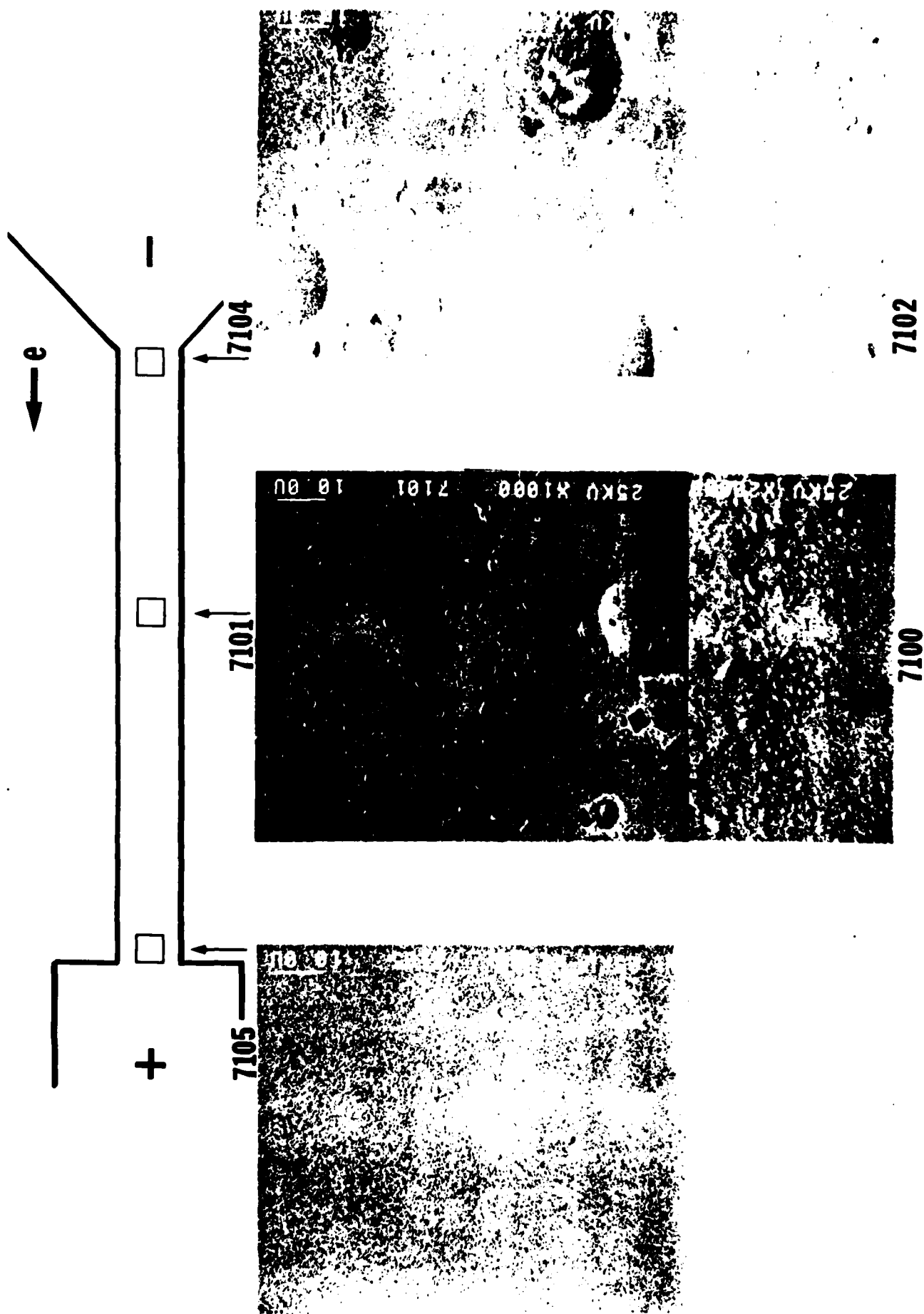
Figure 2. Sample GAu8E, gold film 815 Å thick on microscope slide which contained Na, DC stressed at $J = 5.72 \times 10^5$ amps/cm^2 on ceramic substrate holder for 237.0 hours to failure.
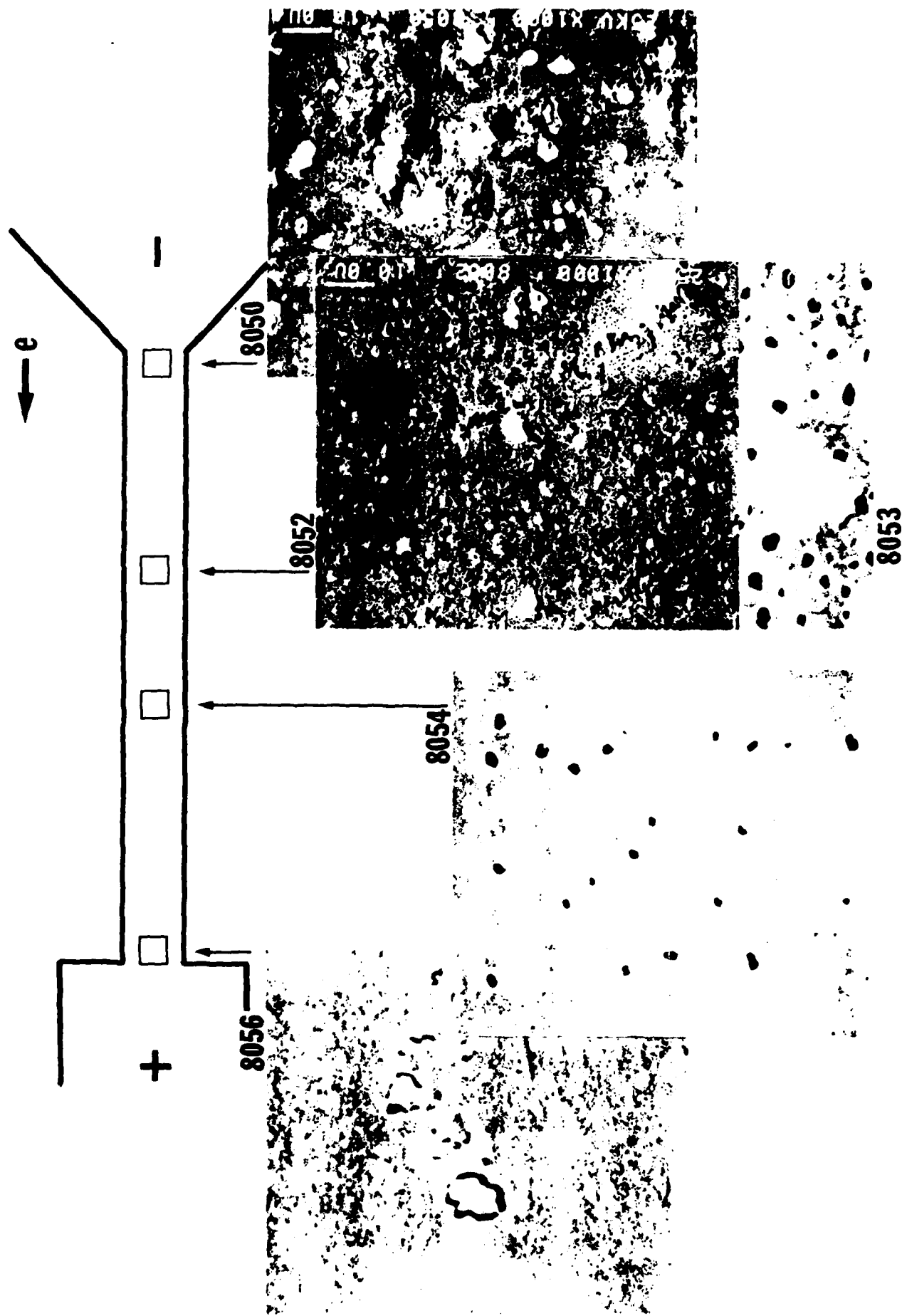
Figure 3. Sample GAu23C, gold film 795 Å thick on microscope slide which contained Na, DC stressed on metal substrate at J = 6.11 x 10^5 amps/cm^2. Sample stressed for 474.0 hours to failure.

limited to that caused by dirt, dust, etc. The highest hole density is relegated to the central portion of the stripe. Since the central surface at higher magnification (SEM micrographs #8053 and #8054) looks comparable to samples subjected to thermal grooving,[3] it is postulated that the predominant failure mechanism for this particular sample was thermal grooving with the superposition of some electromigration-induced damage. Two classes of holes are apparent (SEM micrograph #8053), either very small or very large. Two distinct hillock types are visible as well, one type being tall and faceted (SEM micrographs #8052 and 8053) and usually associated with the large holes. The other type is a platelike hillock that is uniformly distributed along the surface (SEM micrographs #8050 and 8052). The holes and hillocks are not asymmetrical, i.e., occurring at either end of the stripe, but rather are associated with one another locally.

Most noteworthy is the high amount of surface material accumulated on the cathode surface (SEM micrograph #8050). The features associated with this roughened surface include:

a) platelike, hexagonal growths

b) roughened areas of whisker-like material

c) several typical electromigration-induced hillocks

Material transport that opposes electron flow is again evident.

An example of an extremely resilient Au sample that was deposited on soda-silicate glass is shown schematically in Figure 4. GAu39A experienced 7.76×10^5 amps/cm^2 for 3382 hours prior to removal from the DC circuit. The long-running time recorded for this sample is not typical for Au films deposited on soda-silicate glass. Holes appear only along the anode region (SEM micrograph #2400) and nucleate from circular patches of material. These
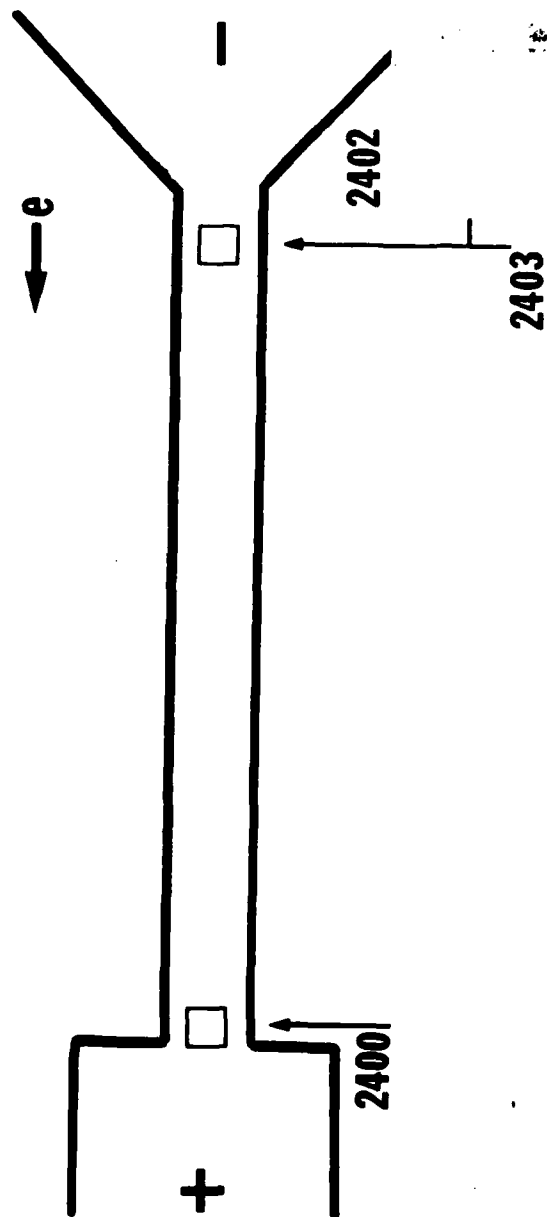
Figure 4. Sample GAu39A, gold film 800 Å thick on microscope slide which contained Na, DC stressed on metal substrate at $J = 7.76 \times 10^5$ amps/cm^2. Sample stressed for 3,382 hours to failure.

patches also appear to nucleate several faceted hillocks as well. The cathode area shows many features (SEM micrographs #2402 and 2403), including:

a) At least two distinct hillock types. In the lower left-hand corner of SEM micrograph #2403 very small, elongated and faceted growths are seen. The second type appears uniformly distributed along the surface and is more flattened, larger and less crystallographic than the first type.

b) Areas of de-adhesion (SEM micrographs #2402 and 2403) are seen only along the cathode region that we have postulated may contain Na compounds that have collected at the film-glass interface.

c) The cathode area shows much more surface roughening than the anode region with aboslutely no hole growth in the cathode region.

In this sample, hole and hillock growth are extremely asymmetric, in that holes appear only on the anode region and material accumulation only in the cathode region. As we have seen before, this directional nature of material transfer clearly opposes the electron flow.

Doping of Na Through the Use of Fused Glass

A fused glass having a different composition than that of soda-silicate glass (see Figure 5) was employed in the next class of samples. The deposition, testing and analytic procedure that was described earlier was maintained here. The sample, after being subjected to 7.85×10^5 amps/cm^2 for 3366 hours, was removed for examination. As seen in Figure 6, the highest hole density appears near the anode area (SEM micrograph #3702). This density decreases (SEM micrograph #3703) and becomes minimal in the central stripe portion (SEM micrograph #3700). Circular patches that often contain large

Composition of Substrate Glasses

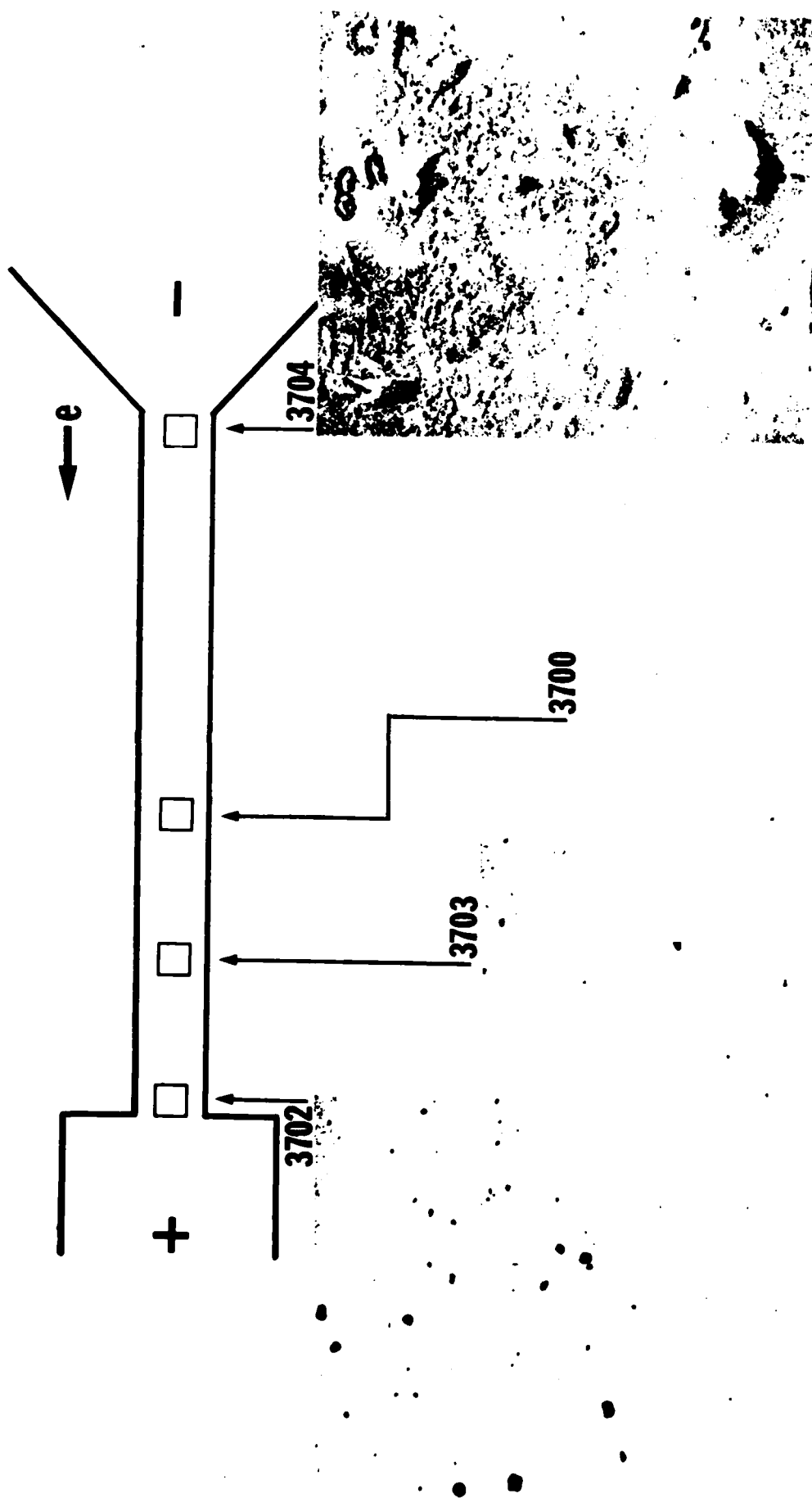| Element	Microscope Glass (Float Glass)	Fused Glass
Si (SiO_2)	72.6%	65.8%
Al (Al_2O_3)	0.18%	9.6%
Ca (CaO)	8.7%	2.6%
Mg (MgO)	3.0%	0.015%
Na (Na_2O	13.7%	9.16%
K (K_2O)	0.065%	3.8%
Fe (Fe_2O_3)	0.074%	0.036%
Ti (TiO_2)	0.008%	0.65%
B (B_2O_3)	---	8.4%

Figure 5

Figure 6. Sample GAu39F, gold film 800 Å thick on glass containing Na, DC stressed at $\bar{J} = 7.85 \times 10^5$ amps/cm^2 on metal substrate holder, for 3,366 hours. Current stressing terminated prior to failure.

faceted growths also nucleate near the anode region (SEM micrograph #3702) and enlarge to a maximum size in the central stripe region (SEM micrograph #3700). Similar "patches" were seen in sample GAu39A on soda-silicate glass. Typical electromigration-induced hillocks appear only at the cathode (SEM micrograph #3704). Again, this hole-hillock configuration represents reversal in mass transport.

It should also be noted that the growth of the circular patches with increasing temperature towards the stripe center indicates that a thermal component must also be added to the mass transport. In contrast to the soda-silicate samples, no "bubbles"—an indication of de-adhesion—were evident in the fused glass samples.

Gold Thin Films Containing Ion-Implanted Na

The final method used for the introduction of Na in gold thin films that was examined in this study was ion bombardment. The facility located at the Murray Hill branch of Bell Laboratories allowed for precise doping of the films in contrast to all of the preceeding methods which produced an infinite source of Na in direct contact with the powered film. Since most glasses contain up to 10% Na_2O, the ion bombarded sample compositions were chosen to contain 50 ppm to 4.83% Na. (This could be attained through the use of ion fluxes that ranged from 3×10^{12} to 3×10^{16} Na^+ ions/cm^2 of Au film.) The films were deposited using standard procedures and then sent to Murray Hill for appropriate doping. After this process, the samples were returned to be tested and analyzed in the aforementioned manner. Control samples were constructed for composition analysis and differential reflectometry confirmed qualitatively that the films contained the appropriate doping levels.

It is important to cite some of the major differences between the processes mentioned earlier and that of ion bombardment. First, it is obvious that Na enters the film at the gold-air interface for the vacuum-doped and ion bombarded films, while Na enters by way of the gold-glass interface during the other methods. This difference could be critical since the Gibbs Free Energy for sodium oxidation is extremely negative at room temperature (approximately 90 kcal/mole). In the vacuum-doped and ion bombarded films, the sodium would oxidize on the film surface, reach stability during that process, and never penetrate the film. For the glass-doped samples, however, the driving force for oxidation could enhance the diffusion of Na from the film-glass interface to the film surface, allowing for the Na to affect electrotransport in Au. Second, the ion bombardment process is a bulk process in which the Na is introduced to the film over a uniform surface. It is well known, for example, that this process is capable of producing non-equilibrium compositions. This ion penetration also causes a tremendous number of point defects in the sample. Ion bombarded samples, then, would contain a uniform distribution of Na as well as a high concentration of point defects. During powering, these defects can either anneal out or act as short circuit paths for the sodium atoms. In contrast, in all of the aforementioned doping techniques—vacuum furnace doping and the various glass slide doping— equilibrium criteria must be met, which require that less than 1% Na is soluble in gold. Any excess Na is relagated to the film's grain boundaries with Na diffusion occurring in those same boundaries. Obviously, this locational difference of the Na impurities alters their effect as scattering centers during the conduction process.

In Figure 7, a 1000 Å Au film containing approximately 1.7% Na (an ion flux of 10^{16} Na$^+$ ions/cm^2) is schematically portrayed. Failure occurred after 6.27 x 10^5 amps/cm^2 for 1320 hours. As noted earlier, electron flow is from right to left and reversal of material transport would dictate material depletion near the anode. The roughened surface produced by Na ion bombardment can be easily appreciated after inspection of SEM micrograph #4301. Crater-like features attributed to actual ion impact are plainly visible. The uniformity of the method can be observed in SEM micrographs #4302 and 4307, where the lower, smooth portion of the sample was masked during the bombardment process. Close examination of these two micrographs shows a high density of small holes often associated with these crater-like features near the anode (SEM micrograph #4307) and an absence of such holes near the cathode (SEM micrograph #4302). This hole formation again opposes electron flow.

Figure 8 describes another approximately 1000 Å Au film that was bombarded with Na ions. This sample received, however, a much lower ion flux of 3 x 10^{13} Na$^+$ ions/cm^2 which represents a doping level of 50 ppm. This sample failed after 6.9 x 10^5 amps/cm^2 after 605.7 hours of powering. As the lower three micrographs verify (SEM micrographs #49, 45 and 41) severe evidence for thermal grooving is visible. Another effect, however, is superimposed upon this rather uniform thermal damage. As in the previous ion bombarded sample, an asymmetric behavior is noted at lower magnification (SEM micrographs #48 and 43). While holes are evident throughout the sample, their density and size have dramatically increased near the anode region (SEM micrograph #48). Again, such behavior opposes electron flow.
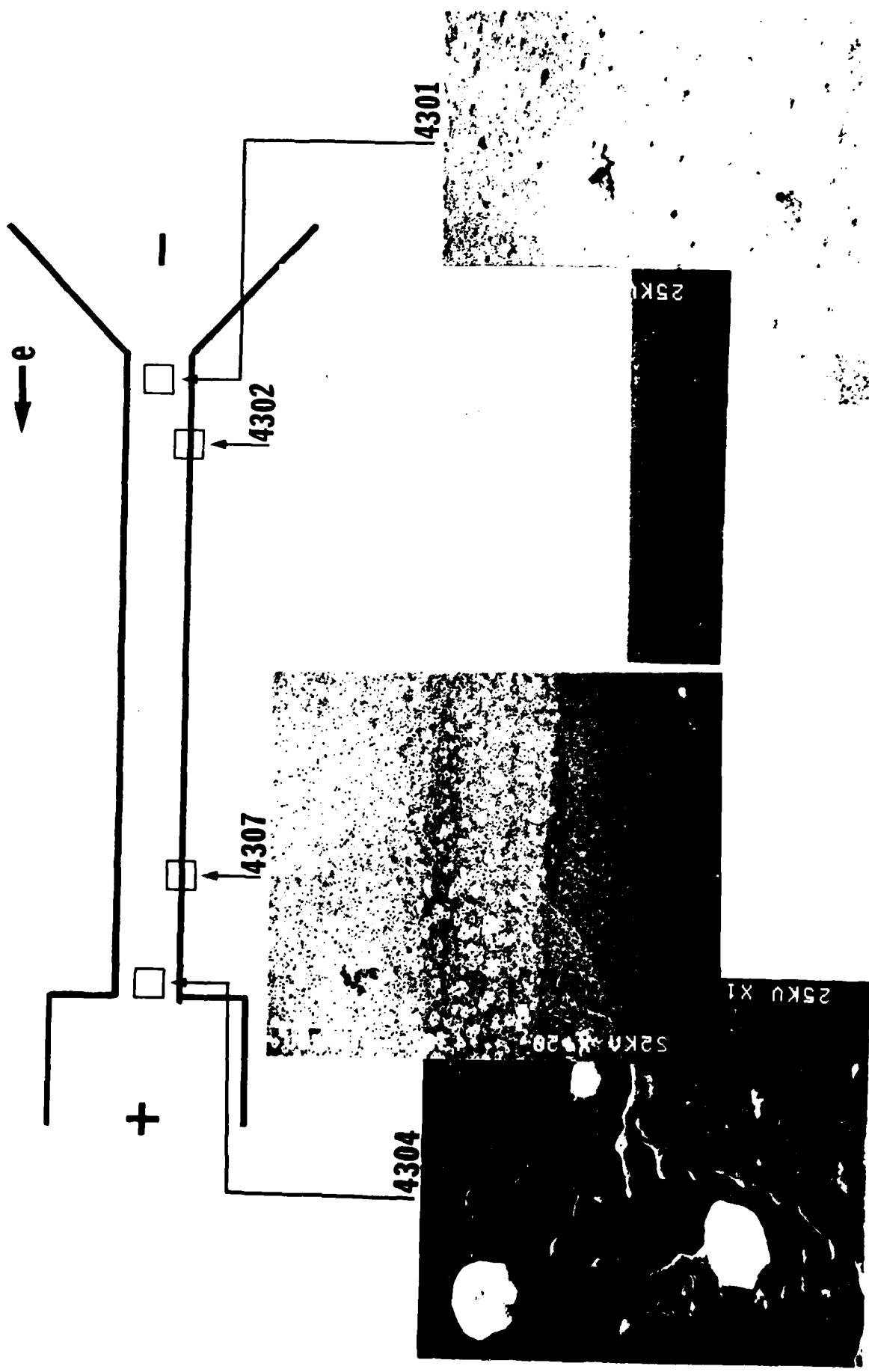
Figure 7. Sample GAu29A, gold film 1001 Å thick containing 1.7% Na (ion implantation flux of 10^{16} Na$^+$/cm^2) on quartz substrate, DC stressed at J = 6.27 x 10^5 amps/cm^2 on metal substrate holder for 1,320 hours to failure.
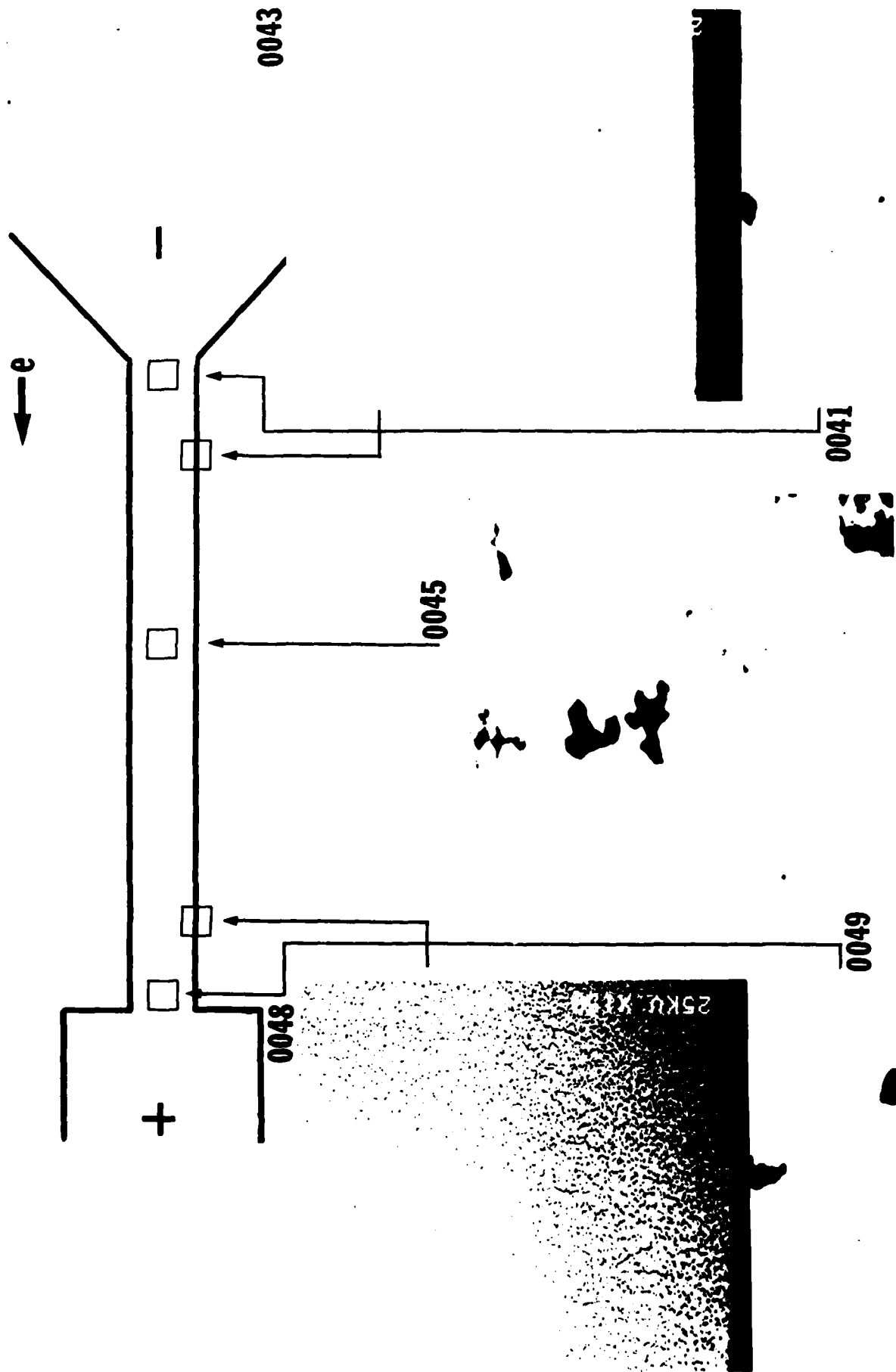
Figure 8. Sample GAu31A, gold film 1007 Å thick containing 50 ppm Na (ion implantation flux of 3×10^{13} Na$^+$/cm^2) on quartz substrate, DC stressed at $\bar{J} = 6.9 \times 10^5$ amps/cm^2 on metal substrate holder for 607.7 hours to failure.

Conclusion

In order to isolate the effect of sodium on the reversal of electro-
migration in gold, four techniques were used to test whether reversal could
be attributed to the introduction of Na into Au films independent of the
doping method. It has been found that evidence of electromigration reversal
in Au with Na occurs whether doping is performed by vacuum furnace diffusion,
diffusion from glass substrates of varying composition or ion bombardment.
Reversal is, therefore, independent of processing conditions. These methods
encompassed impurity levels that ranged from 50 ppm to approximately 30% Na,
and reversal was consistently noted. It is apparent, therefore, that minute
concentrations of Na severely alter gold electromigration. These observations
have major implications for the manufacture and reliability of thin gold films.

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Personnel

Chief investigators during the entire contract period (1/1/76-8/31/81) were Dr. Rolf E. Hummel and Dr. Robert T. DeHoff, both professors of the Department of Materials Science and Engineering at the University of Florida.

The main part of the early investigations were carried out by Mr. Bruce Krumeich who graduated in June, 1978 with a Master of Science in Engineering degree. His thesis title was "Solute Addition Effects on the Electrotransport Failure Site in Thin Gold Films."

In September, 1978, Ms. Susan Matts-Goho, a new graduate student, joined the electrotransport project. Susan has a Bachelor's degree in physics and worked for two years in the electronics industry. She has been working since then on the electromigration project and expects to graduate with a Ph.D. in 1982.

In December, 1978, Mr. Mike Goho joined the group. He also has a Bachelor's degree in physics and is likewise pursuing his studies towards a Ph.D. degree.

During a short time period (early 1978 through Fall, 1979), Mr. Phillip Russell worked on the project. He is now at the Solar Energy Research Institute in Golden, Colorado.

In January, 1979, Dr. H. B. Huntington, Department of Physics, Rensselaer Polytechnic Institute, Troy, New York, was visiting our group for one month as part of a Center of Excellence grant provided by the State of Florida. Professor Huntington is internationally known for his contributions to the theory of diffusion and electrotransport in metals and alloys.

Dr. W. Kleinn, a professor at the Institute of Technology in Karlsruhe (Germany) spent one month in 1980 at the Materials Science Department

to interact with our group. Dr. Kleinn in known for his electron microscopy studies on electrotransport in thin films.

Scientific Papers

1. "Electromigration in Thin Films," by R. E. Hummel, Proceedings of the Intl. Conf. on "Electro- and Thermotransport in Metals and Alloys," Niagara Falls, NY, Sept. 22, 1976, AIME, Inc., NY (1977).

2. "The Effect of Minor Constituents on the Electrotransport-Induced Failure Site in Thin Gold Films," R. E. Hummel, B. K. Krumeich and R. T. DeHoff, Appl. Phys. Lett., $\underline{33}$, 960 (1978).

3. "Thermal Grooving, Thermotransport and Electrotransport in Doped and Undoped Thin Gold Films," R. E. Hummel, R. T. DeHoff, S. Matts-Goho and W. M. Goho, Thin Solid Films, $\underline{78}$, 1 (1981).

4. "The Role of Sodium on the Electrotransport Behavior in Thin Gold Films," S. Matts-Goho and W. M. Goho, to be published.

Scientific Interaction

1. Dr. Hummel gave an invited paper on "Electrotransport in Thin Films" at the Technische Universität, Berlin, Department of Metallphysik on July 1, 1977.

2. Dr. Hummel gave an invited paper on "The Direction of Ion Flux in Thin Computer Metallizations" at the Universität Göttingen on August 9, 1977.

3. Dr. Hummel gave an invited paper on "Electromigration in Metals and Alloys, at the Universität Aachen, July 10, 1977.

4. Dr. Hummel presented an invited paper on "Electrotransport in Thin Films" at the Seventh Annual Symposium on Applied Vacuum Science and Technology, held February 13-15, 1978 in Tampa, Florida.

5. Dr. Hummel presented our recent results on electrotransport during his visit on November 2, 1978 to the Max Planck Institute for Materials Research in Stuttgart, Germany.

6. Both Drs. DeHoff and Hummel attended the metallurgical conventions in St. Louis, Missouri in October, 1978 , and in Denver, Colorado, in February, 1978 and had interactions there with scientists working in similar fields.

7. Dr. Hummel gave an invited paper at the Department of Applied Physics, University of Tübingen (Germany) in November, 1979 entitled "Electro-transport in Thin Films."

8. Both Drs. Hummel and DeHoff attended the 108th AIME Annual Meeting from February 18-22, 1979 in New Orleans and had interactions there with scientists working in similar fields.

9. Dr. Hummel gave an invited lecture entitled "Electrotransport in Metals and Alloys" at the Physics Department, University of Florida on Jan. 19, 1981.

10. Dr. Hummel gave an invited paper entitled "Elektrotransport in Metallen" for the "Gesellschaft Deutscher Chemiker," on March 19, 1981 in Aachen, Germany.

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